

BULLETIN

OF THE INSTITUTE OF METALS

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PART 18

INSTITUTE NEWS

The Institute of Metals (Platinum) Medal

The Institute of Metals (Platinum) Medal for 1953 has been awarded by the Council to Professor GEORG MASING, of the Institut für allgemeine Metallkunde, Universität Göttingen, Germany, in recognition of his outstanding contributions in the field of metallography.

The medal, which is put at the Council's disposal by The Mond Nickel Company, Ltd., is awarded annually in recognition of outstanding services to the non-ferrous metal industries, whether on the industrial or scientific side, without distinction of race or country.

The Rosenhain Medal

The Rosenhain Medal for 1953 has been awarded by the Council to Dr. CHARLES ERIC RANSLEY, of the Research Laboratories, The British Aluminium Company, Ltd., Chalfont Park, Gerrards Cross, in recognition of his outstanding experimental and theoretical work on gas-metal equilibria.

The medal, which is put at the Council's disposal by Imperial Chemical Industries, Ltd., is awarded annually in recognition of outstanding contributions in the field of physical metallurgy made by persons under the age of 45 years, irrespective of nationality, sex, or membership of the Institute.

Copper Pass Awards

The Copper Pass Awards, which are made annually from a sum of £200 placed each year at the disposal of the Councils of the Institution of Mining and Metallurgy and of the Institute of Metals, by the Directors of Copper Pass and Son, Ltd., Bristol, are intended to encourage the publication of papers on processes and plant used in extraction metallurgy and on the subject of assaying in the *Transactions of the Institution of Mining and Metallurgy*, and of papers on processes and plant used in the fabrication of non-ferrous metals in the *Journal of the Institute of Metals*.

Provided that the Adjudicating Committee is satisfied that the quality and numbers of papers submitted, of the types for which the awards are made, reach a suitable standard:

(a) £100 per annum is available for one or more Awards to the authors of papers on some aspect of non-ferrous extraction metallurgy, including assaying;

(b) £100 per annum is available for one or more Awards to the authors of papers relating to some process or plant used in the extraction or fabrication of non-ferrous metals.

The Adjudicating Committee meets each year to consider papers published during the calendar year in the two journals referred to above.

The MSS. of suitable papers should be sent for consideration of the respective Publication Committees, addressed to the Editor of one or other of the two journals in the normal way.

1953 Spring Meeting: Visits to Works

Below are given some details of The Pyrene Co., Ltd., to whose works a visit is to be paid on Thursday, 26 March.

The Pyrene Company, Ltd., Brentford

The Pyrene Company was founded in 1914 to manufacture the well-known Pyrene fire extinguisher. The Company rapidly expanded its interests in fire and safety engineering, and has pioneered equipment and methods which have revolutionized fire-fighting technique in many fields; the development of foam branch-pipes and generators and proteinous foam-making compounds for oil-tank fires and aircraft crash fires, is one example. Besides the main factory at Brentford, there are now three subsidiary factories, one situated locally and the other two in South Wales. The central laboratories are at the main factory in Brentford, and it is there that the greater part of the research and development are carried out in all the Company's activities.

New fire hazards, necessitating the development of completely new extinguishants and techniques, are continually arising in modern industry. One recent example is the development of equipment and chemicals for the extinction of metal fires such as those due to magnesium, aluminium, sodium, &c. Much new equipment in the specialized field of aircraft fire safety, such as smoke and flame detectors, can be seen in the Aircraft Research Laboratory.

Apart from its fire and safety activities, the Company early developed other purely engineering interests and, among many other products, has manufactured motor-car bumpers since their adoption in this country many years ago. A feature of the Brentford factory is the quantity production of high-quality electroplated articles of relatively large size from steel through all the stages of pressing, pretreatment, electroplating, polishing, and assembly.

The Metal Finishing Division of the Company deals with a wide range of pretreatment and finishing processes for many metals. The best known of these are the Parkerizing and Bonderizing ranges, which the Company introduced to this country in the late nineteen twenties. Originally the Bonderizing processes were designed to provide corrosion-resistance and paint adhesion. Now, in addition, specially developed variations of them are also used to assist in the cold deformation of metals; e.g. in tube-drawing, wire-drawing, deep-drawing,

PERSONAL NOTES

and cold extrusion. The most recent addition to the Bonding processes for cold-forming is one for stainless steel. Besides their extensive use for protection against corrosion, some Parkerizing processes have also found application in anti-scuffing treatment on gears, tappets, valve guides, camshafts, &c., and a special process developed for this purpose, Parco-Lubrizing, is now in wide use for reducing wear on working parts, in the automotive industries. Examples of all these applications will be seen in the Company's laboratories.

Election of Members

The following 11 Ordinary Members, 1 Junior Member, and 22 Student Members were elected on 31 December 1952:

As Ordinary Members

- ABDO, Ahmed Fathi, Head, Mechanical Department, Technical Secondary School, Cairo, Egypt.
ANSTEY, John Henry, B.Sc., Patent Department, The Mond Nickel Co., Ltd., Sunderland House, Curzon Street, London, W.1.
BLIGH, Ernest W., Metallurgical Engineer, S. E. Opperman, Ltd., Boreham Wood, Herts.
BOOTH, Ernest K., B.Sc., Materials Engineer, A. V. Roe (Canada), Ltd., Gas Turbine Division, Box 430, Terminal "A", Toronto, Ont., Canada.
FAST, Johan D., Chief Metallurgist, Research Laboratories, N. V. Philips' Gloeilampenfabrieken, Eindhoven, Holland.
GEORGE, Wilbert R., Student of Metallurgy, University College, Swansea.
GOLDA, Edward W., B.Sc., Technical Supervisor, Los Angeles Tube Division, Phelps Dodge Copper Products Corporation, 6100 Garfield Avenue, Los Angeles 22, Calif., U.S.A.
JOHANSSON, Klas-Erik, Chief Metallurgist, Uddeholms Aktiebolag, Hagfors Järnverk, Hagfors, Sweden.
LAMBILLIOTTE, André Alphonse Joseph Emile, Administrateur-Directeur, Société Anonyme des Usines Gilson, La Croyère, Belgium.
WARLAND, Eugène, Ing. Civil des Mines, Administrateur-Directeur, Société Anonyme Métallurgique de Prayon, Trooz, Belgium.
WELDON, James W., 2315 Harrison Street, Kansas City 8, Mo., U.S.A.

As Junior Member

- SKINNER, Colin, B.Sc., Student of Metallurgy, University College, Cardiff.

As Student Members

- AL-SAMARRAI, Shakir Haj Khalaf, Student of Metallurgy, University College, Swansea.
BROWN, Darryl Anthony, Student of Metallurgy, Cambridge University.
DAVIES, Peter Malcolm, Student of Metallurgy, University College, Swansea.
EMMERSON, Leslie Gordon, Student Metallurgist, Stewarts and Lloyds, Ltd., Bilston, Staffs.
FISHER, Raymond John, Metallurgical Apprentice, Stewarts and Lloyds, Ltd., Bilston, Staffs.
GOODCHILD, Keith Trevor, Metallurgical Trainee, Stewarts and Lloyds, Ltd., Bilston, Staffs.
GRANT, John Michael Seafeld, Student of Metallurgy, Cambridge University.

- HEATON, Herbert Barrie, Student of Metallurgy, The Technical College, Bradford.
HUME, Gerald James Thomas, Metallurgical Apprentice, Stewarts and Lloyds, Ltd., Bilston, Staffs.
LLOYD, Brian Arthur, Metallurgical Apprentice, Stewarts and Lloyds, Ltd., Bilston, Staffs.
MARKOWICZ, Jan Henryk, Student of Metallurgy, Battersea Polytechnic, London, S.W.11.
MODLEN, Geoffrey Frank, Student of Metallurgy, Cambridge University.
PARKER, Robert, Metallurgical Apprentice, Stewarts and Lloyds, Ltd., Bilston, Staffs.
PINNEY, Victor, Student of Metallurgy, Liverpool University.
SMITH, Michael Duncan, B.Met., Student of Metallurgy, Sheffield University.
SMITH, Robert, B.Sc., Spectrographer, Johnson, Matthey and Co., Ltd., 78 Hatton Garden, London, E.C.1.
STOLARCZYK, Janusz Eugeniusz, Student of Metallurgy, Battersea Polytechnic, London, S.W.11.
SZKOPIAK, Zygmunt Czeslaw, Student of Metallurgy, Battersea Polytechnic, London, S.W.11.
TABERNER, James Norman, Student of Metallurgy, College of Technology, Manchester.
THOMAS, Raymond William, Foundry Metallurgist, Dartmouth Auto Castings, Ltd., Dartmouth Road, Smethwick 40, Staffs.
THOMPSON, Stewart Philip, Student of Metallurgy, Cambridge University.
WESTROPE, Alan Richard, Student of Metallurgy, University College, Swansea.

PERSONAL NOTES

MR. W. BARR has been elected Honorary Treasurer of the Iron and Steel Institute.

MR. J. BATEMAN has left Hard Metal Tools, Ltd., Coventry, and taken a post with Furth-Sterling, Inc., Pittsburgh, Pa., U.S.A.

MR. L. J. CARTMELL has joined the staff of Henry Wiggin and Co., Ltd., Birmingham.

MR. G. J. CATTERALL has been awarded the degree of B.Sc. with Second Class Honours of London University and the A.R.S.M. He has now taken up an appointment with Group Four Metals, Ltd., London, E.14.

MR. J. B. CHAPPELL has taken a post at the Waunarlwydd Works of Imperial Chemical Industries, Ltd., Metals Division, South Wales.

DR. W. F. COXON has been appointed Managing Director of Arrow Press, Ltd., Watford, publishers of a number of technical journals.

MR. N. H. G. DANIELS has left England for the United States, where he is to undertake research at the Institute of Engineering Research, University of California, Berkeley, Calif.

DR. C. H. DESCH has been elected an Honorary Member of the American Society for Metals.

MR. P. G. DODD has been appointed Foundry Technical Officer to Austral Bronze Pty., Ltd., Alexandria, N.S.W.

MR. C. R. FAULKNER has left Birmingham University to take up an appointment in the Ministry of Supply at Culcheth, near Warrington.

DR. G. E. GARDAM, who until recently was Director of Research of the Design and Research Centre for the Gold, Silver, and Jewellery Industries, has taken up an appointment with Aluminium Laboratories Limited, Banbury.

MR. R. H. HANNAFORD has taken up an appointment in the Research Laboratories of Fairey Aviation Co., Ltd.

DR. HAROLD HARTLEY, Chairman of Radiation, Ltd., Birmingham, has been awarded the Melchett Medal of the Institute of Fuel.

MR. T. L. JOHNSTON has left Liverpool University, where he was recently awarded the degree of Ph.D., and is now at the Atomic Energy Research Establishment, Harwell.

PROFESSOR P. J. LACOMBE has left the Laboratoire Central des Traitements Chimiques at Vitry (Seine) and has been appointed Professor of General Metallurgy and Director of the Centre de Recherches Métallurgiques in the Ecole Nationale Supérieure des Mines, Paris, in succession to Professor Crussard.

PROFESSOR R. H. MYERS has been elected to the Council of the Australian Institute of Metals (Sydney Branch).

MR. J. R. PARISH has left Elliott Brothers, Ltd., Boreham Wood, and is now with Cathodic Corrosion Control, South Audley Street, London.

MR. R. PARKER, of Aluminium Laboratories, Ltd., has been transferred from Geneva to Banbury.

SEÑOR JORGE A. SABATO has now returned to the Argentine after a visit to Great Britain. He wishes to thank the many members of the Institute who assisted in making his visit successful.

PROFESSOR CYRIL STANLEY SMITH, Director of the Institute for the Study of Metals at the University of Chicago, is planning to visit various metallurgical research centres in England and Western Europe in February and March.

Death

The Editor regrets to announce the death of:

MR. JOHN STANLEY BOWDEN, B.Met., on 28 November. He had been a Senior Metallurgist on the staff of the Tin Research Institute since the end of 1946.

NEWS OF LOCAL SECTIONS AND ASSOCIATED SOCIETIES

London Local Section

At a meeting of the Section held at the Royal School of Mines, London, S.W.7, on 6 November, Professor G. V. RAYNOR, M.A., D.Sc., Professor of Metal Physics at Birmingham University, gave a lecture on:

An Approach to the Theory of Ternary Alloys

The lecturer said that though further work was necessary to extend the present theory of binary alloys to solvents other than those of the first two groups of the Periodic Table, sufficient was known about alloy formation in copper and silver to justify an approach to the theory of ternary alloys based on these metals. The influence of known factors upon the details of ternary equilibrium diagrams could be traced, with particular reference to the shapes of the primary solid-solubility isothermals, and to the extent to which binary electron compounds projected into the ternary equilibrium model. In particular, a simple thermodynamical analysis

was possible for solubility isothermals at which the primary solid solution was in equilibrium with a stable intermetallic compound. It could be demonstrated that there was a close similarity between the influence of the atomic size-factor on the crystal structures of binary $3/2$ electron compounds, and its influence on the structures of the corresponding ternary phases.

These considerations could be used to interpret, and in favourable cases to predict, the homogeneity ranges of close-packed hexagonal and body-centred cubic $3/2$ electron compounds in ternary systems, provided that no very stable intermetallic compounds between the two solute metals interfered. The influence of stable compound formation was illustrated by a comparison of the isothermal diagrams for the Ag-Mg-Zn, Ag-Mg-Sn, and Ag-Mg-Sb alloys. In the first case, the Mg-Zn compounds were of low heat of formation and exerted no influence on the equilibria in silver-rich alloys. The compound Mg_2Sn , of higher heat of formation, entered into equilibrium with the $3/2$ electron compounds in the system Ag-Mg-Sn, and Mg_3Sb_2 , of still higher heat of formation, prevented equilibrium between the $3/2$ electron compounds in the system Ag-Mg-Sb, and itself entered into equilibrium with the primary silver-rich solid solution. The existence of ternary compounds had to be allowed for in developing the theory, and work was in progress to define the conditions under which they might be expected. Investigation of the Cu-Al-Sn and Cu-Al-In systems had indicated that such compounds might occur where any tendency to ordering in a binary intermediate phase was enhanced by the inclusion of a third metal. This led to interesting structural relationships between binary and ternary phases.

Scottish Local Section

At a meeting of the Section held in Glasgow on 10 November, Mr. R. A. F. HAMMOND lectured on:

The Properties and Engineering Applications of Electrodeposits

The lecture was primarily concerned with heavy nickel and hard chromium deposits as used in the engineering industries for salvaging worn or over-machined parts or for hard-surfacing.

After briefly reviewing the history of the subject and its present-day applications, the lecturer gave some information on the economics of the process. The techniques and equipment are similar to those employed in decorative electroplating, but the great importance of obtaining very strong adhesion and good mechanical properties of the deposits calls for conscientious and skilful workmanship. Much ingenuity is frequently required to secure reasonably uniform distribution of the thick deposits commonly employed in this class of work, for example, by the use of auxiliary anodes or cathodes.

Strong adhesion of the electrodeposit to the basis metal is of paramount importance. Given suitable cleaning and depositing conditions, the bond between the deposit and the base is atomic in nature, and slides were shown of photomicrographs of sections of electrodeposited metals in which the grain boundaries of the base were continued into the electrodeposit. This is possible for pairs of metals belonging to different crystallographic systems and for lattice spacings differing by as much as 15%. In such circumstances the adhesion of the deposit may equal or even exceed the tensile strength of the component metals.

Adhesion of, or approaching, this order is possible on most

low-alloy structural steels and on many other metals and alloys used in engineering. For other materials, e.g. certain light alloys, special cleaning methods are necessary, and the adhesion is liable to be somewhat weaker; on a few materials only very weak adhesion is obtainable.

In addition to being strongly adherent, the electrodeposit must have suitable mechanical properties. The hardness of electrodeposits commonly exceeds that of the corresponding metal in the as-cast or rolled condition, and moreover the mechanical properties, including the hardness, can be controlled over a considerable range by adjusting the depositing conditions. The diamond pyramid hardness of electrodeposited nickel as normally applied ranges from 180 to 400 with U.T.S. values of 28 and 55 tons/in.² and elongations of 35 and 6% respectively. Electrodeposited chromium has a diamond pyramid hardness usually ranging from 750 to 950 and virtually no ductility.

Most electrodeposited metals undergo a change in volume after deposition which sets up an internal stress in the deposit. In general, the stress is contractile, but some deposits, notably zinc and lead, show compressive stress. Nickel and chromium deposits are normally strongly contractile, but certain bright nickel deposits exhibit a slight compressive stress. The residual stress in these electrodeposits does not usually lead to practical difficulties, but may cause a marked reduction in the fatigue limit of the steel base, which could be serious in those limited applications in which alternately stressed components operate near the critical loading.

Heat-treatment of chromium-plated components between 150° and 300° C., as commonly employed for relieving hydrogen embrittlement, has been shown to cause a still further reduction in the fatigue limit of the steel, and the advantage of using a higher temperature, e.g. 440° C., as described by Logan, and also methods of minimizing the effect of fatigue-limit reduction by shot-peening and other means, were discussed.

Finally, a brief account was given of the behaviour of electrodeposited nickel and chromium as bearing surfaces.

OTHER NEWS

Conferences on "The Deep Drawing of Metals" and "Continuous Casting of Ingots"

The Department of Industrial Metallurgy, University of Birmingham, is arranging its annual two-day Conference this year on Monday and Tuesday, 16 and 17 March. The subjects will be "The Deep Drawing of Metals", on 16 March, and "Continuous Casting of Ingots", on 17 March. Full details of the programme may be obtained from the Secretary, Department of Industrial Metallurgy, The University, Edgbaston, Birmingham 15. No fees are chargeable for the attendance, but registration is required.

DIARY

Local Sections and Associated Societies

- 10 February. South Wales Local Section.** "Dislocations in Crystals", by Dr. B. A. Bilby (University College, Metallurgy Department, Singleton Park, Swansea, at 6.30 p.m.)
- 12 February. Liverpool Metallurgical Society.** "Nucleation in Metals and Alloys", by J. H. O. Varley. (Liverpool Engineering Society, The Temple, Dale Street, Liverpool, at 7.0 p.m.)

- 16 February. Sheffield Local Section.** "The Constitution and Properties of Some Titanium-Base Alloys", by W. A. Baker. Joint meeting with the Sheffield Society of Engineers and Metallurgists and the Sheffield Metallurgical Association. (The University, St. George's Square, Sheffield 1, at 7.30 p.m.)
- 18 February. Manchester Metallurgical Society.** "Metallurgical Aspects of Lubricating Problems", by A. L. H. Perry. (Engineers' Club, Albert Square, Manchester, at 6.30 p.m.)
- 25 February. Liverpool Metallurgical Society.** Visit to The British Aluminium Co., Ltd., Warrington.
- 27 February. Birmingham Local Section.** All-Day Symposium on "Making the Best of Metals". Opening address by R. Lewis Stubbs. (College of Technology, Suffolk Street, Birmingham, at 10.15 a.m.)
- 27 February. North-East Metallurgical Society.** "Steel Foundry Radiographic Practice", by G. M. Michie. (Cleveland Scientific and Technical Institute, Middlesbrough, at 7.15 p.m.)
- 2 March. Sheffield Local Section.** Annual General Meeting, followed by "The Economic Use of Non-Ferrous Metals", by F. Hudson. Joint meeting with the Institute of British Foundrymen, Sheffield Branch. (Sheffield College of Commerce and Technology, Department of Engineering, Pond Street, Sheffield 1, at 7.30 p.m.)
- 3 March. Oxford Local Section.** "John Dalton and Modern Thought", by Stephen Toulmin. (Black Hall, St. Giles, Oxford, at 7.0 p.m.)
- 3 March. South Wales Local Section.** "Recent Advances in Furnace Design", by E. S. W. Eardley. (University College, Metallurgy Department, Singleton Park, Swansea, at 6.30 p.m.)
- 4 March. Manchester Metallurgical Society.** "Precipitation-Hardening", by Dr. H. K. Hardy. (Engineers' Club, Albert Square, Manchester, at 6.30 p.m.)
- 5 March. Birmingham Local Section.** "Copper and Copper-Alloy Development", by Dr. E. Voce. (James Watt Memorial Institute, Great Charles Street, Birmingham 3, at 6.30 p.m.)
- 5 March. Leeds Metallurgical Society.** "Lubrication in Cold-Working Processes", by A. E. McAulay. (Chemistry Department, The University, Leeds 2, at 7.15 p.m.)
- 5 March. London Local Section.** "Dislocations in Crystals" by Dr. B. A. Bilby. (4 Grosvenor Gardens, London, S.W.1, at 7.0 p.m.)

Other Societies

- 12 February. Institute of Metal Finishing, North-West Branch.** "The Electrodeposition of Tin and Its Alloys", by Dr. J. W. Cuthbertson. (Engineers' Club, Albert Square, Manchester, at 7.30 p.m.)
- 16 February. Institute of Metal Finishing, London Branch.** "Roughness of Nickel Deposits", by C. J. Swanson. (Northampton Polytechnic, St. John Street, London, E.C.1, at 6.0 p.m.)
- 18 February. Society of Chemical Industry, Corrosion Group.** "Some Recent Advances in Corrosion Research", by Dr. W. H. J. Vernon. (The Stephenson Building, King's College, Newcastle-upon-Tyne, at 2.30 p.m.)

For Appointments Vacant, see p. a, facing Abstracts 448.

NEW TECHNIQUES OF METALLURGICAL RESEARCH *

B3

INTRODUCTION

By PROFESSOR A. G. QUARRELL,† D.Sc., Ph.D., A.R.C.S., F.I.M.

THE topic chosen is one with a very wide appeal. The industrial metallurgist feels that at a Symposium of this nature he may learn of methods of detecting defects in his product before too much work has been put into it, or may hear of a new technique that can be adapted to control one of his processes. The research metallurgist knows that a new technique may speed up a research many times, or even make possible what could not previously be done at all.

It has been said that most of the great advances in science have been associated with the introduction of new techniques. This may be going too far, but every really original development must widen our horizon and enable us to concern ourselves directly with phenomena formerly hidden from us. Techniques are therefore of the utmost importance in research at every stage: in data collecting, when the working hypothesis is being formulated, and again when the hypothesis is being subjected to experimental test. Indeed, a research may be held up for a long time while a suitable means of conducting the crucial experiments is being worked out. This usually happens when the technique required is novel in a qualitative sense.

Advances in technique may be regarded as qualitative or quantitative. The qualitative advance makes available information of an entirely new kind, whereas quantitative advances enable us to know more exactly what could have been known approximately with existing techniques.

Good examples of the former are provided by metallography and X-ray crystallography. The introduction of metallography enabled men to see for the first time how crystals were arranged in metals and the different phases that were present. The later development of X-ray methods provided a means of determining how atoms are arranged within crystals. Both of these were qualitative advances, and both have greatly improved our understanding of the nature and properties of metals.

Improvements of technique in the quantitative sense may also have beneficial effects, for knowledge that is insufficiently precise is usually confusing. Indeed, many of the laws of science could not be elucidated until techniques of sufficient accuracy and sensitivity had been worked out. If a graph is drawn to represent the growth of metallurgical knowledge with time, it will consist of a series of slopes separated by abrupt steps. The steps represent the "qualitative" developments in techniques, whereas the slopes correspond to the steady improvement that goes on almost continuously. One gains the impression that these slopes are getting steeper

and that the advances gained through the improvement of existing techniques are becoming ever more important.

We should all like to know what will mark the next really big step. Speculation is probably idle, but it may lie in the full exploitation of the possibilities opened up by radioactive isotopes. Much more development work needs to be done in this field, but, in principle at least, we are now able to follow the movements of different kinds of atoms within the crystal lattice—something not previously possible.

Such discoveries are, by their nature, few and far between, but there is still plenty of scope for the improvement of existing techniques. Two of the papers to be discussed are concerned with metallography, illustrating that the basic technique which has formed the metallurgist's most powerful method of investigation for almost ninety years is still capable of improvement. Considerable advances have taken place in this field during the last few years; they include phase-contrast microscopy, the fuller application of polarized light, multiple-beam interference methods, the examination of surface profiles by means of tapered sections, and I suppose we could also include microhardness testing. Much work has aimed at improving the methods of preparing metals for microscopic examination, particularly with regard to the time taken. Thus, in principle, the methods used by Sorby to examine polished and etched specimens in 1864 were identical with those in use to-day, but the times taken to polish a specimen were much longer. Now the time required to prepare a satisfactory specimen is measured in minutes, whereas it took a whole week to produce a surface to satisfy Sorby. However, his standards were very exacting.

In their student days metallurgists are required to draw the structures they see through their microscopes. This method is valuable because it ensures that detailed consideration is given to the structures observed, but I wonder how many metallurgists realize that even in the early years of the present century this was the only method by which a microstructure could be recorded. Photography has come to play a tremendously important role in modern metallurgy, and it would probably not be going too far to assign to it the importance of a step on the graph of progress. Spectrography, cinematography, high-speed cinematography, metallography, X-ray crystallography, radiography, are all possible in their modern form only because of the photographic technique, and there can be no doubt that in its various forms it has greatly speeded up the acquisition of new knowledge.

* Papers forming the basis of a Symposium organized by the Birmingham Local Section of the Institute of Metals, and held in

Birmingham on 29 February 1952.

† Professor of Physical Metallurgy, University of Sheffield.

Mention of photography serves to remind us that many of the techniques we use were introduced for quite different purposes and have been subsequently adapted for metallurgical work. To-day, workers in all branches of science are on the look-out for new techniques that will help them to solve outstanding problems or enable them to obtain information more quickly or more precisely, and they realize that developments in quite a different field may provide them with the method they are seeking. One beneficial effect of this is that in an age of increasing specialization, in which it is becoming increasingly difficult for a worker in one subject to understand and keep abreast of developments in another, they share a general interest in techniques, and scientists of many types meet to discuss common problems. This is well illustrated by the various groups of the Institute of Physics.

Some of the principles that can be used in devising new techniques are so complex that a new career, that of "technique specialist", seems to be growing up. In a sense he is a scientific mechanic, who, when he is told what requires to be done, will work out a suitable method. Having done so, he has no further interest in it, except that the experience

he has gained will help him to tackle somewhat similar jobs with greater confidence and efficiency in the future. I think it is fair comment that the "technique specialist" generally produces a solution which is far from simple and which requires elaborate and expensive apparatus.

One of the outstanding changes in our research laboratories in the last twenty years is the great increase in quantity and in complexity of the apparatus used. While recognizing that the days of sealing-wax and string have gone for ever as far as metallurgy is concerned, I hope we shall not go too far in the other direction and come to regard complexity of technique and elaborate apparatus as essential to research.

Let us judge a technique, not in terms of the skill shown by the man who developed it, but by what that technique will enable us to do and by the new horizons it opens up, and let us put a premium on simplicity. The simpler the better, so long as it is capable of doing the job that is to be done; a complicated technique is justified only if it is quite impossible to do the same thing more simply. Our discussion should help in achieving this object, for simplification and improvement come from the free interchange of ideas among men with common interests.

THE USE OF DIAMOND DUST FOR METALLOGRAPHIC POLISHING

By L. G. TOTTLE,* A.R.Ae.S., L.I.M.

Historical Survey

THE use of diamond dust as a polishing medium for jewels and hard-metal carbides has been well known for some time, but its application to the preparation of metallographic specimens is comparatively recent. Woodside and Blackett¹ in 1947 described the use of diamond dust for polishing specimens of molybdenum carbide and cast molybdenum, previously prepared by surface grinding on a diamond-impregnated wheel. Tarasov and Lundberg² in 1949 demonstrated how it might be used for specimens of high-speed steel which had first been hand polished on a diamond hone in place of the conventional emery cloth.

More recently attention has been drawn by Perryman³ to the use of diamond dust for polishing metallographic specimens. He described the preparation of specimens of galvanized coatings, nickel + chromium-plated brass and aluminium-silicon alloy, oxide films on copper, and hard inclusions in aluminium-base alloys. The specimens were prepared in the usual manner by grinding down to 4/0 emery paper before being polished on a Selvyt pad impregnated with diamond dust. The diamond dust had a particle size of 0.1 μ , and the Selvyt pad, moistened with white spirit, was used on a special disc. The disc had a raised rim and revolved at a fairly slow speed, so that the diamond dust should not be thrown off the pad and wasted.

Preliminary Treatments

In the research laboratories of The Mond Nickel Co., Ltd., a very large number of ferrous and non-ferrous micro-

specimens, covering a considerable range of hardness and having a wide variety of structures, have to be prepared each week. The floor space available for metallographic polishing is limited, and therefore it has been necessary to introduce new techniques with a view to reducing the time required for preparing each specimen before micro-examination. In addition to the use of diamond dust as a medium in the final stages of polishing, wet grinding on rotating emery-paper discs and automatic polishing have accordingly been introduced.

For most of the microspecimens, rubbing by hand on emery paper has been discarded. After flat surfaces have been obtained on a grinding machine, they are held by hand on a succession of silicon carbide papers of different degrees of coarseness, attached to 7½-in.-dia. horizontal discs revolving at 520 r.p.m. A water-resistant type of paper is used, which is flooded with water during the polishing operation, this serving to keep the specimen cool and to wash off any loose particles of abrasive powder. The cutting action of these papers is very swift indeed, and a specimen may be prepared from a surface-ground finish to 600 grit (approximately 4/0), suitable for pad polishing, in under 2 min. Two of the polishing wheels are shown in Fig. 1, the left-hand one being ready for use and the other opened for changing the paper disc. A special jig has been developed to enable three ½-in.-dia. specimens, or more of smaller diameter, to be prepared at the same time. An assembled jig containing three specimens and the component parts of a jig are shown in Fig. 2.

For final polishing on rotating pads, automatic polishing

* Technical Assistant, The Mond Nickel Co., Ltd., Research and Development Laboratory, Birmingham.

machines, modified slightly from the design introduced by The British Non-Ferrous Metals Research Association, have been installed (Figs. 3 and 4). In this type of machine the specimen is traversed mechanically across the face of the rotating pad, while being turned through approximately 90° at each traverse. The pressure on the specimen may be varied at will, but is controlled and is uniform throughout the polishing operation. The time required to polish a specimen mounted in Bakelite, or multiple specimens in the special jig, is about 15 min. At the present time four of these machines are operating almost continually.

Diamond-Dust Experiments

Many of our samples, e.g. those which had been coated with zinc or nickel, complex alloys, &c., could not be polished

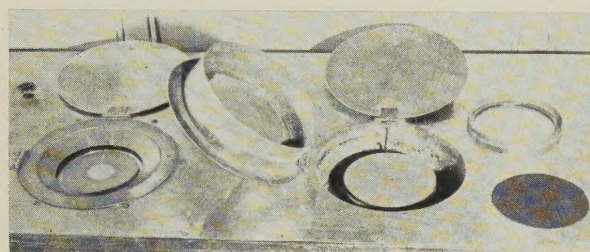


FIG. 1.—Equipment for Grinding on Wet Silicon Carbide Papers.



FIG. 2.—Jigs for the Polishing of Bar Samples.

satisfactorily in a reasonable time by the established abrasives, so that the publication of Perryman's work led to an immediate investigation of the potentialities of diamond dust.

The initial experiments showed that satisfactory results could be obtained on $7\frac{1}{2}$ -in.-dia. rimless wheels rotating at 190 r.p.m. Speeds up to 520 r.p.m. are now in use, without noticeable loss of diamond dust. No special apparatus has therefore been required.

About 0.025 g. of diamond dust is first rubbed into the dry Selvyt cloth attached to the wheel, and it is then sparingly lubricated with paraffin or anhydrous alcohol. Needless to say, the pad is not washed out after use, but is carefully preserved in a stoppered bottle if it has to be taken off the wheel. Diamond dust has been used for the polishing of specimens by hand and on the automatic polishing machines with very good results.

The following are a few examples where diamond-dust polishing has proved particularly valuable. Galvanized cast-iron samples are difficult to polish by conventional methods.

Fig. 5 (a) shows a carefully prepared section polished on γ -alumina. Staining and considerable differences in levels of the zinc, ferrite, and graphite are apparent. Fig. 5 (b) shows a flat unstained field obtained by the use of diamond dust. The surface is, however, covered with very fine scratches, and this has been found to be a feature of diamond-dust polishing. These scratches can be removed by a polish of short duration on light magnesia or γ -alumina.

It is frequently necessary to prepare specimens of corroded and scaled materials with a view to examining the interface between the deposit and the base material. These deposits differ considerably in hardness from the body of the sample and, when conventional techniques are used, they are often worn down or broken away. By diamond-dust polishing satisfactory specimens are usually obtained at the first attempt,

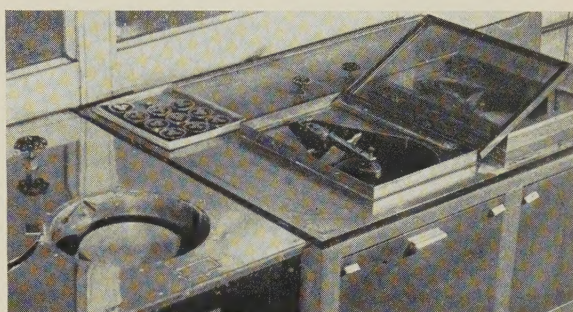


FIG. 3.—Modified Automatic Polishing Machines.

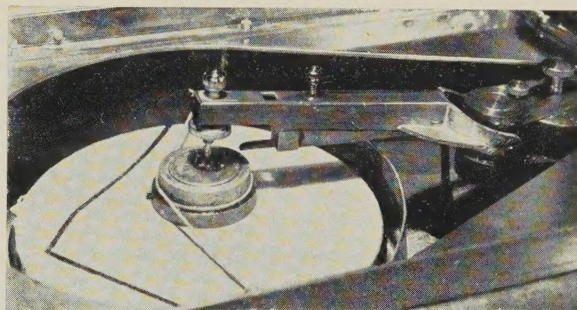


FIG. 4.—Close-up of Modified Automatic Polishing Machine.

thus saving a great deal of time and energy and requiring less skill on the part of the operator. Fig. 6 is a photograph of a section from a corroded nickel-chromium alloy bearing an oxide deposit. This specimen was prepared from the rough, i.e. including grinding operations, in 22 min.

In Fig. 7 (a) is shown an oxide film on a Nimonic alloy. This specimen was prepared for polishing by depositing nickel over the oxide film to protect the edge of the section. It was polished with care on alumina, but the oxide film is badly damaged. Fig. 7 (b) illustrates a similar specimen prepared on diamond dust. In this sample the section has been mounted in Bakelite without extra protection of the edge and has been polished quite successfully. The time which would be spent in plating the specimen has thus been saved, and a better result achieved.

Complex nickel-base alloys are difficult to prepare by normal means without the introduction of considerable relief or without pulling out some of the constituents. They are also difficult to etch successfully, so that they have to be

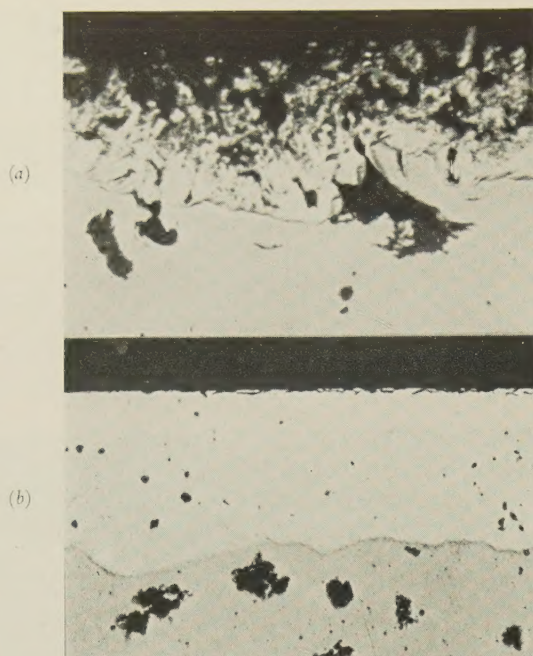


FIG. 5.—Galvanized Nodular-Graphite Cast Iron. Unetched.
× 500.

(a) Polished on γ -alumina. (b) Polished on diamond dust.

examined unetched with the aid of phase-contrast illumination. By this method of illumination only very slight differences in level can be tolerated, and it is for this type of specimen that diamond-dust polishing proves especially useful. Fig. 8 is a photograph of an alloy containing three phases differing considerably in polishing and etching characteristics, which has been photographed in the unetched condition, using phase-contrast illumination.

For joining high-temperature-resistant materials, precious-metal brazing alloys have been developed. Fig. 9 shows a

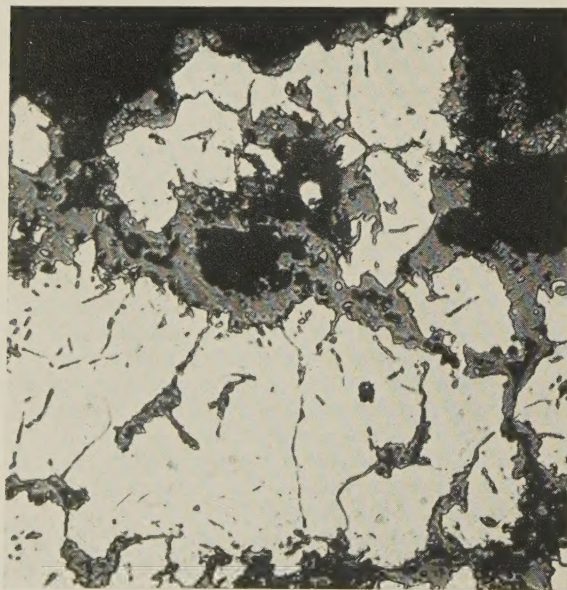


FIG. 6.—Corroded Nickel-Chromium Alloy. × 400.

deposit of one of these alloys on Nimonic. Once again there is a considerable difference between the polishing characteristics of the base material and the brazing alloy, but the photograph shows that a satisfactory flat field has been obtained right across the interface between them.

As a final example, the profiles of metal powders are easy to examine by transmitted light, but it is their cross-sections which are often most informative. When such powders are mounted in finely sieved Bakelite and polished first on fine papers and then on diamond dust, a good specimen may quickly be obtained. Fig. 10 is a photograph of an iron powder produced from iron wire by means of a metallizing pistol. Oxide skins on the particles and small globules of oxide within them may be clearly seen.

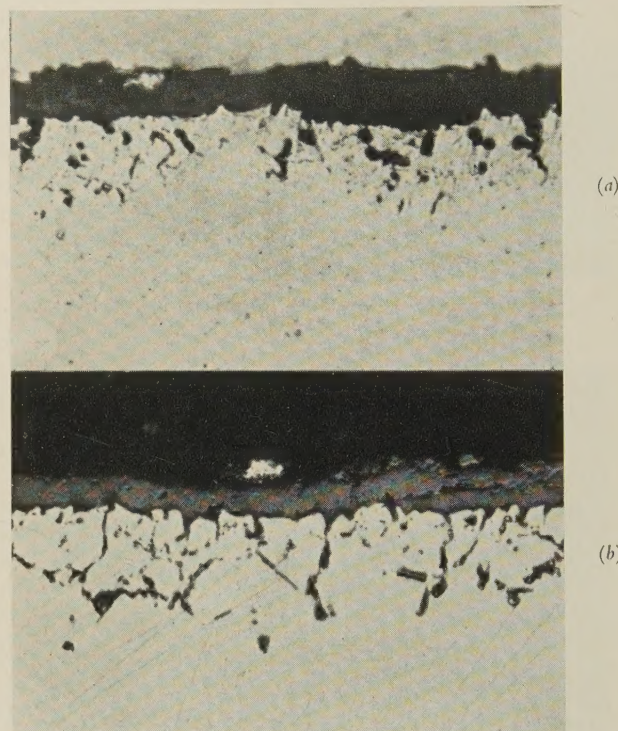


FIG. 7.—Oxide Films on Nimonic Alloy. × 1000.

(a) Polished on γ -alumina. (b) Polished on diamond dust.

Many other applications have been found for the diamond-dust polishing technique, but space does not permit of further illustration.

Advantages of Diamond Dust

It is probable that the versatility of diamond dust as a polishing medium is due to its great hardness, which allows it to cut through particles of widely differing hardness at much the same rate and enables the abrasive particles to retain a good cutting edge for a considerable time. Electron-micrographic examination of the diamond dust shows it to have a sharply angular form such as is illustrated in Fig. 11 for powder of $4-8\ \mu$ particle size. The uniform particle size of the diamond dust undoubtedly assists greatly towards the quality of the finish produced.

A disadvantage associated with the use of diamond dust as a polishing medium is that, because of its great cutting power, all samples show a network of fine scratches. These

do not hinder low-power examination, but for high-power examination or for photography they must be eliminated by a further short period of polishing on alumina or light magnesia. The duration of this treatment must obviously be a compromise between the desire to obtain a scratch-free field and to preserve flatness and absence of objectionable

The initial cost of diamond dust is fairly high, but so very little is required and it lasts for so many samples, that on these grounds alone it becomes a useful proposition. When time saved in the preparation of otherwise difficult specimens is taken into consideration, however, it becomes most attractive.

This paper has attempted to show how simple it is to in-

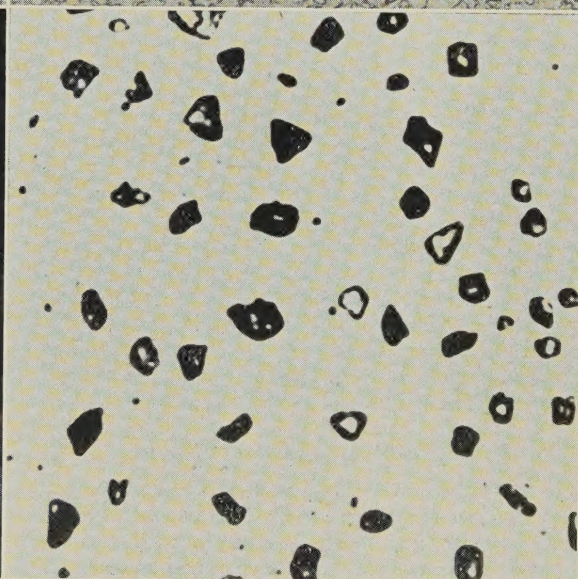
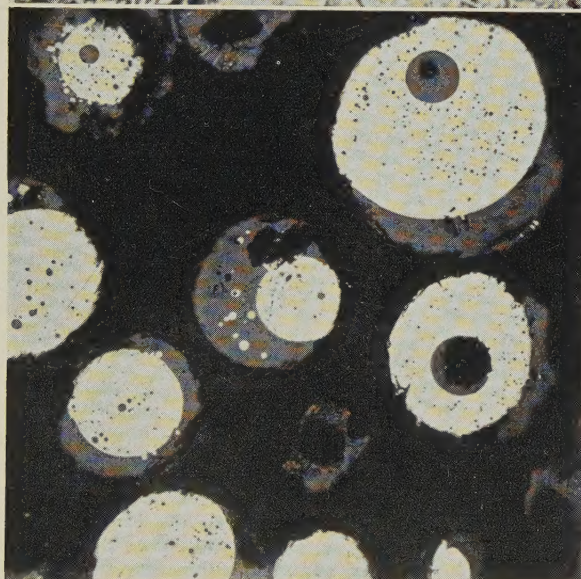
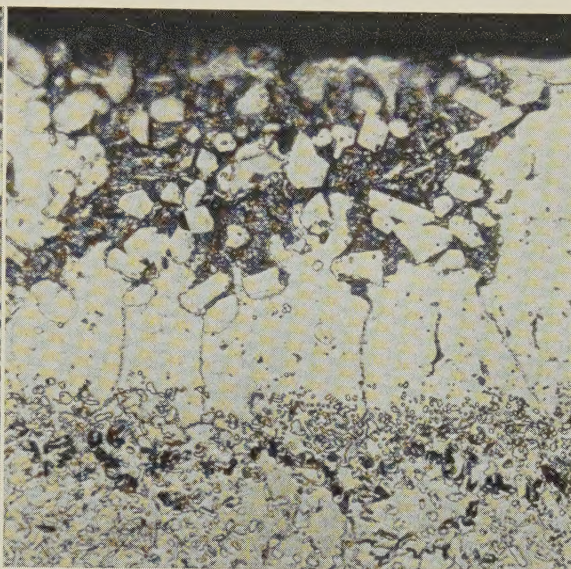
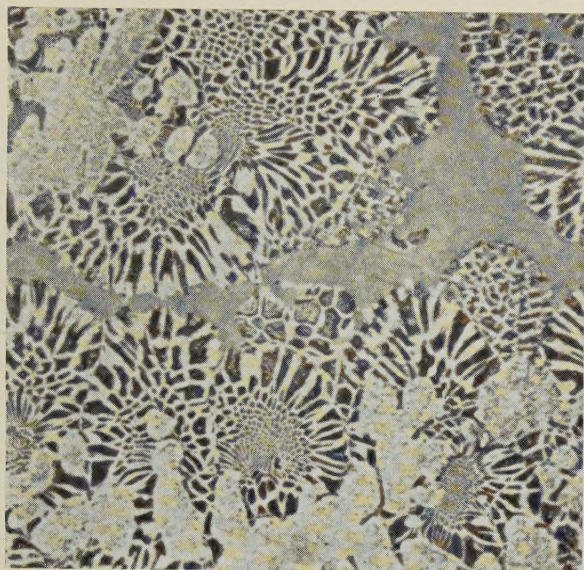


FIG. 8.—Complex Nickel-Base alloy. Photographed, unetched, by phase-contrast illumination. $\times 250$.

FIG. 10.—Iron Powder. $\times 400$.

FIG. 9.—Deposit of Precious-Metal Brazing Alloy on Nimonic. $\times 400$.

FIG. 11.—Diamond Dust, Particle Size 4–8 μ . $\times 1000$.

relief in the sample. From the point of view of freedom from scratches, the final finish is therefore not always equal to that obtained by highly skilled operators on materials for which specialized techniques have been evolved. The main advantages of diamond-dust polishing lie in the good finishes that can be obtained by less highly skilled polishers in short periods of time and the flatness of fields containing phases of widely differing hardness.

Introduce the diamond-dust technique using normal equipment. All that is necessary is a supply of diamond dust, preferably of the finest grade and of uniform particle size, a Selvyt pad, and a supply of paraffin or other light-oil lubricant. The speed of the pad is not critical, provided that the dust is not flung off as it revolves, and this may be avoided by rubbing the diamond dust into the pad.

It is understood that supplies of polishing paste, in which

the diamond dust (of 0-2 μ size) is held in a petroleum-jelly base, are on the market in this country. In America devices similar in action to the familiar grease-gun are available, so that the amount of paste to be applied may be carefully controlled.

Acknowledgements

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USES OF THE HOT-STAGE MICROSCOPE

By P. J. E. FORSYTH,* A.I.M.

Problems Involved in High-Temperature Microscopy

THE microscopical examination of hot metal surfaces involves a number of factors which tend to lower the resolving power of the optical system. The main problem is that of obtaining a large working distance and yet retaining a useful degree of resolution. Naturally, the examination of a hot metal surface by any optical instrument necessitates a limiting safe working distance and a protective cooling system for the front lens. As the specimen will be enclosed in a small furnace, an observation window is necessary, which adds to the optical limitations. In spite of these detrimental factors the definition that can be obtained even with simple optical systems, is sufficient to make high-temperature microscopy a useful technique.

It is not proposed to deal here with these optical systems, but one of the most useful developments has been that of the reflecting microscope objective which possesses the great advantage of a large working distance.

Apart from the optical defects which may be encountered while working at elevated temperatures, the retention of a metallographic surface finish is important, and staining or oxidation must be avoided. This will require a controlled atmosphere within the furnace or heating *in vacuo*.

Interpretation of Observations

Some atmospheres can affect the micro-appearance of the metal. Shuttleworth, King, and Chalmers,¹ while investigating grain-boundary movements in silver, found that striations were visible on the surface when oxygen was present, which were not observed in a nitrogen atmosphere. At this point it must be emphasized that one is examining a free surface of the metal, and the surface appearance should not necessarily be taken as indicative of the underlying structure. This point is well illustrated in Fig. 1, which would seem to show boundary films of a second phase. The structure is that of a leaded brass which has been heated to 500°C. During re-cooling it appeared as if the grain boundaries were being penetrated by the lead. On repolishing, however, it was found that the lead had only migrated along the surface grooves produced by the thermal etching of the boundaries.

A similar case is shown in Fig. 2. This specimen is an aluminium-8% silver alloy which was electropolished and then heated to 300°C. It was found on repolishing that the γ phase had existed only as a thin film on the surface, and this is not indicative of the amount of the phase present in the alloy. Thus the surface appearance, e.g. striations, boundary penetration, and the presence of a second phase, does not necessarily represent the true structure of the metal, as the structure is often influenced by the energy associated with the free surface and that of the boundaries meeting the surface.

Applications of Technique

An application which is receiving much attention at the moment is to the study of recrystallization and boundary migration. This phenomenon lends itself well to hot-stage microscopy, as the boundaries in the metal usually show up quite clearly after heating. This may be due to more rapid volatilization of the metal in the boundary regions or to the boundary tension producing a groove on the surface. If the boundary moves to a new position, a new groove is formed, and the old groove may persist or completely heal. There is no doubt that surface migration of the metal atoms plays an important part in this healing process. The persistence of the old boundaries is often useful, as it reveals the amount of migration that has occurred. This persistence may be due to loss of material by volatilization, to staining, or even to precipitation. Fig. 3 shows a surface where boundaries have persisted owing to staining and the temporary retention of the grooves, and Fig. 4 illustrates precipitation where the boundary meets the surface. Fig. 3 also shows small craters where low-melting-point particles have liquated. In fact, the whole grain-boundary network was observed to liquefy before the grains themselves. This suggests another potential use for the technique, i.e. the detection of small quantities of constituents or impurities of a lower melting point than the matrix. This is especially useful if the impurities exist as boundary films which may be hard to detect by normal microscopic methods. As soon as a constituent or region of segregate liquates, this can be detected, because the molten liquid sinks or forms a meniscus and appears to the observer as a small crater or groove.

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Another line of investigation would be to employ the hot-stage technique together with a mechanism for stressing the specimen while under observation. The combination of the two techniques should be useful in the study of creep phenomena. An apparatus was built at the R.A.E. to study the effect of stress on boundary migration. A small ribbon of zinc was electropolished and then heated while being stressed. It was quite easy to follow the boundary move-

It is interesting to note that Figs. 4 and 6 provide evidence of the intermittent migration of grain boundaries.

Conclusions

The hot-stage technique for examining metallographic specimens may be very useful for investigating recrystallization and boundary migration, and for detecting segregation in

FIG. 1.—A Leaded α -Brass, Showing Boundary Films of Lead Existing at the Surface Only. $\times 500$.

FIG. 2.—Aluminium-8% Silver Alloy, Showing Surface Precipitation. $\times 1000$.

FIG. 3.—Grain-Boundary Residual Markings in Zinc, and Surface Craters Where Impurities Have Melted. $\times 100$.

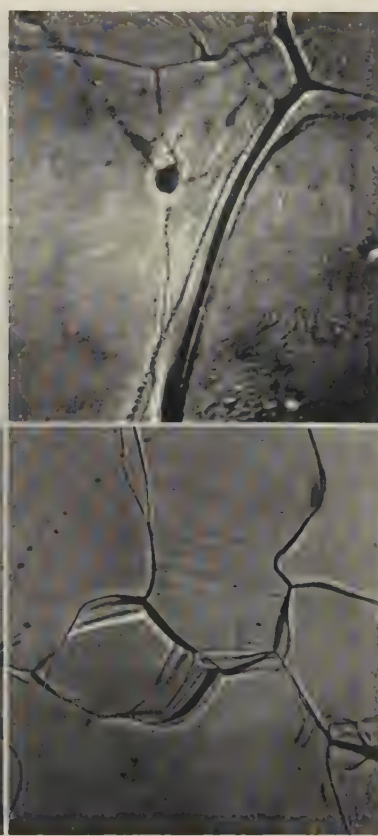
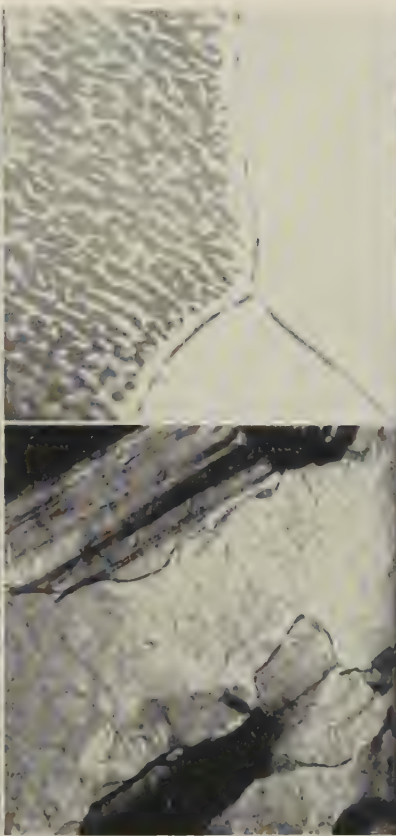
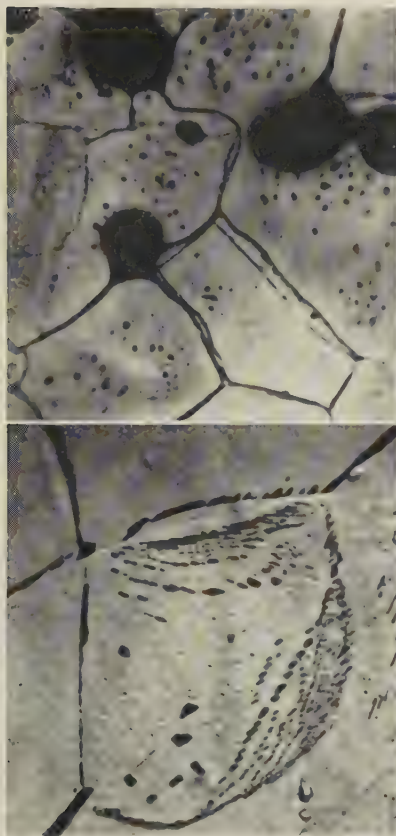


FIG. 4.—Surface Precipitation in Aluminium-8% Silver Alloy at Regions Where Grain Boundaries Meet the Surface. $\times 1000$.

FIG. 5.—Moving Grain Boundaries Observed in Zinc at 350° C. $\times 100$.

FIG. 6.—Boundary Positions in Zinc Delineated by the Flow That Has Occurred in Each Position. $\times 1000$.

ments at a magnification of $\times 250$ (16-mm. objective) with very little loss of definition. Fig. 5 shows a moving grain boundary as seen at 350° C. Any form of deformation which produces differences in surface level can be examined continuously, i.e. it does not require etching to reveal the extent of the microstructural changes, and therefore is very suitable for the hot-stage technique. This is illustrated in Fig. 6, which is of a zinc specimen subjected to creep stresses at 350° C. The boundary positions at different time intervals are not in this case revealed by thermal etching or by precipitation, as in previous examples, but by the boundary slip that has occurred when the boundary was in each position.

alloys, low-melting-point impurities such as boundary films, &c. The technique may also prove valuable for examining solid-phase transformations which produce constituents of a different colour or form, or if there is a bulk change that will involve relief of the constituents. However, the limitations of the technique must not be overlooked, particularly as the complicating factor of the free surface may lead to erroneous observations and conclusions.

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THE USE OF GEIGER COUNTERS IN X-RAY-DIFFRACTION STUDIES

By G. K. WILLIAMSON,* B.Sc., Ph.D.

Introduction

X-RAY diffraction has been for many years a well-established technique in many branches of metallurgical research. Much of its initial success was due probably to the simplicity of the equipment necessary to give qualitative results, which enabled the techniques to be tested without excessive expenditure. In very few laboratories are X-ray studies now carried out using the simple equipment originally installed. Precision-made multi-purpose cameras have very largely replaced the sealing wax and retort stand, and more reliable automatic X-ray sets the simple but unstable gas tubes. It is not surprising, therefore, that the recording of X-ray intensity by means of films is being replaced by direct detection, using Geiger counters and similar devices.

Much work has been done to explore the possibilities of Geiger counters, although their acceptance and usage is fairly limited, particularly in Europe. Counters are superior to films in most applications of interest to the metallurgist, although it is unlikely that they will ever supersede films in cases where a two-dimensional pattern is required, as in the Laue method. The following review may help potential users to gauge the advantages of this new technique for their own problems.

The Principles of Counters†

Geiger counters (and also proportional counters) are a logical development from the ionization chamber used in many of the classical X-ray studies. X-ray photons, entering any ionization chamber, produce ionization during their absorption. Approximately $10^2 \pm$ ion pairs/photon are

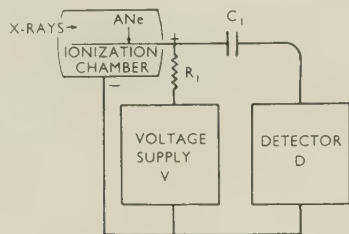


FIG. 1.—The Basic Circuit for Ionization Chambers and Counters. In the case of self-quenching Geiger counters R_1 should be in the range 1–10 megohms.

produced, and these drift towards the electrodes, where they collect, giving up their charge of about 10^{-17} coulombs. Since ionization chambers have a capacity of the order of 10^{-11} farads, there is a voltage change on the electrodes of approximately 10^{-6} V./photon. If the ionization chamber were used in the manner shown diagrammatically in Fig. 1, where a voltage V is maintained between the electrodes sufficient merely to prevent recombination of the ion pairs,

any sudden changes in the voltage between the electrodes would be transmitted to the detector D by the condenser C_1 (the resistance R_1 being made sufficiently large to prevent the charge leaking away too quickly). In principle, the detector D could be used to count each ionizing event, but the difficulties of amplifying a voltage “pulse” of 10^{-6} V. renders this impracticable. It is possible, however, to make the ionization chamber do some of the amplification merely

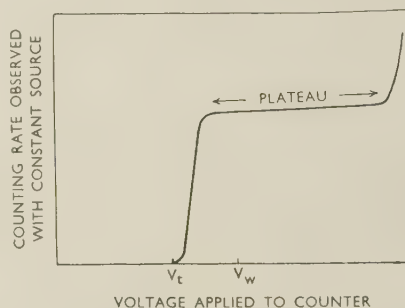


FIG. 2.—A Typical Geiger-Counter Plateau Curve. The plateau usually has a slight slope of less than 0.1% change in response per volt, necessitating some stabilization of V_w , the working voltage.

by raising the voltage V . At a certain value of the voltage field the electrons, during their acceleration towards the anode, may acquire sufficient energy to produce further ionization, so that the initial N ion pairs result in NA electrons being collected, producing a voltage pulse of $NA \times 10^{-8}$ V.; A is called the gas amplification.

A chamber working under these conditions is called a proportional counter, since the output pulse is proportional to the energy of the ionizing radiation up to values of A of about 10^4 (Hanna *et al.*⁶). By the use of complicated circuits discrimination against unwanted radiation is possible (Curran *et al.*⁷; Arndt and Riley⁴; Lang⁵).

At still higher fields the gas amplification begins to “saturate”, until finally, in the Geiger region, approximately 10^9 ion pairs are produced, irrespective of the initial number due to the ionizing event. This region is determined by plotting counting rate against applied voltage, and a plateau, two or three hundred volts long, is found where the response is substantially constant, as in Fig. 2. The working voltage V_w is at the lower end of the plateau, usually about 80 V. above the threshold voltage V_t . Depending on the counter design, this voltage may lie anywhere in the range 500–2500 V. The Geiger region is useful because the voltage pulse per ionizing event is approximately 10 V., and this can be detected and counted with little or no amplification by robust and stable circuits. Although it is the Geiger counter with which this paper deals, many of the remarks apply equally well to proportional counters.

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† For fuller descriptions of the mechanism of counting see Korff,¹ Wilkinson,² and Sharpe and Taylor.³

‡ The number of ion pairs is a function of the energy of the ionizing radiation. For a fuller discussion, see Arndt and Riley⁴ and Lang.⁵

Counters usually have cylindrical geometry, consisting of a tubular cathode and concentric fine-wire anode. For X-rays the counter is filled to a pressure of about one atmosphere with a noble gas,* usually argon, together with small additions of a polyatomic gas. The X-rays enter through a thin window of glass, mica, or aluminium, usually parallel to the counter axis, though in some designs it is perpendicular.

After an ionizing event an avalanche of electrons falls on the central wire, leaving a sheath of positive ions, which moves relatively slowly towards the cathode. This ion sheath reduces the field to a value below the threshold (and is responsible for the saturation of the gas amplification), thus rendering the counter totally insensitive to further ionizing events for a period known as the "dead time", usually about 10^{-4} sec. The counter slowly recovers its sensitivity until the positive ion sheath reaches the cathode, when it becomes fully sensitive once more. Another electron tends to be released with this neutralization, causing the cycle of events to be repeated, unless the release is suppressed or "quenched". The addition of traces of a polyatomic

gas, results in a great increase in the stability of the counter and in reliable and reproducible calibration. One of the serious effects which may result if external quenching is omitted is a violent increase in plateau slope (Parratt and Hempstead⁹), and there is little point in avoiding the use of this type of circuit for any but the most qualitative applications.

Circuits for Use with Quenched Counters

(a) The Voltage Supply (V in Fig. 1)

A variable voltage of 500–2000 V. at a few micro-amp. is generally required, and although dry batteries can be used, a mains-driven power pack is less hazardous. Some form of stabilization is essential, since the response of a Geiger counter to a constant source of X-rays is not wholly independent of the voltage. Plateau slopes of 0.1%/V. are common, particularly for old counters and high counting rates, unless a quenching circuit is used. For the counter to be stable to within 1%, the power pack must be stable to within 0.5% or better for the maximum mains fluctuations. Commercially available circuits have more than adequate stability. (Proportional counters require an extremely high stability if they are to be used for wave-length discrimination.)

(b) The Detector (D in Fig. 1)

One simple form of detector used by Trost¹³ is a galvanometer placed in series with the counter so as to measure the mean current flowing. The current flowing is discontinuous, owing partly to pulse produced by each photon and partly to the random arrival of photons; thus the galvanometer must have a long period in order to give a reasonably steady reading and must be sensitive, since even high counting rates produce a total current of only about one micro-amp. Such a detector is not normally used, although it would certainly suffice for many qualitative applications, and it is generally replaced by a counting-rate meter employing some A.C. amplification and with provision for a variable "period" (or integrating time constant) and a variable sensitivity. Many excellent circuits are commercially available, a number of them including a stabilized power pack, and the details need not be described here. An alternative form of detector is one where each voltage pulse is counted. Electromagnetically operated veeder counters, such as a post office register, are suitable for slow counting rates of less than 300/min. (High-speed mechanical counters are capable of speeds of 5000/min.) However, for very high counting rates it is usual to scale down the pulses electronically by a factor of 100 or more before supplying the mechanical register. Such a scaling unit is capable of counting at speeds of up to 10^6 counts/sec.

The choice between the two forms of detector is not always an easy one. The counting-rate meter can easily be made to produce a record of changing intensities by coupling it to a recorder, such as is used for recording temperature fluctuations. This is a great asset, since it enables many types of scanning to be done automatically. The use of direct counting, on the other hand, is inconvenient for use with changing intensities, although devices have been produced to give automatic recording (Lang¹⁴). The use of a scaling circuit is usually much slower than the use of a counting-rate

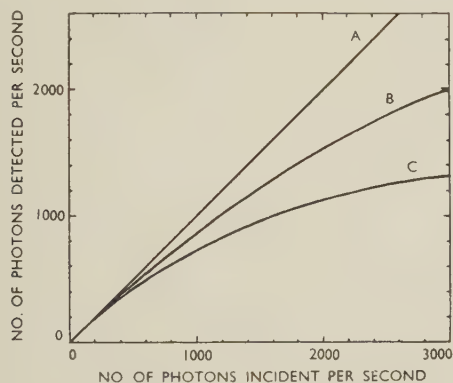


FIG. 3.—The Effect of Counting Losses on the Responses of Counters. Curve A is the ideal linear response (obtained at these rates with proportional counters). Curve B is the response of a Geiger counter with a 200 μ sec. dead time to a non-fluctuating source of X-rays. Curve C is the response of the same counter operated from a pulsed source, such as a self-rectifying X-ray tube running on A.C. The response is linear within 10% for counting rates of up to 2000/sec. for B and 1000/sec. for C.

gas makes the counter self-quenching at lower counting rates, but it is better practice to reduce the anode voltage below V_t by a quenching circuit. If this "imposed dead time" is greater than the recovery time of the counter, the stability, the plateau slope, and the life of the counter are greatly improved.

Since their arrival is random, some photons may enter the counter when it is insensitive and be lost. For qualitative work these losses become serious at high counting rates, as shown in Fig. 3, and the counter must either be calibrated, or a correction made for these losses[†] (Schiff¹⁰; Blackman and Michiels¹¹; Hall and Williamson¹²).

A commercially available quenching circuit, which has a predetermined and very stable imposed dead time allowing accurate counting-loss corrections, has been described by Cooke-Yarborough *et al.*⁸, and its use, for the reasons given

* Krypton is often used to obtain a high efficiency for detecting $\text{MoK}\alpha$ radiation.

† The effective dead time for counters with no external quenching is a function of the detecting-circuit sensitivity, and this is rarely

stable and frequently depends on the rate of count. Proportional counters have negligible dead-time losses up to rates of 10^6 counts/sec., and this is one of their principal advantages.

meter, but a considerably greater accuracy is possible. In order to understand the reason for this greater accuracy, it is necessary to discuss the statistics of counting and the basic functioning of a counting-rate meter.

The Statistics of Counting

Photons do not arrive at regular intervals, but in a random fashion. Thus if N counts per unit time are observed, this will rarely coincide with the average counting rate which

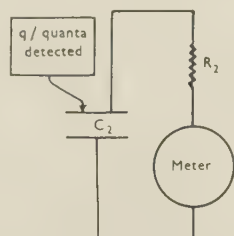


FIG. 4.—The Basic Circuit of a Counting-Rate Meter. Under steady-state conditions the meter reads a current Nq where N is the mean number of counts/sec. If the detection then stops, the meter falls exponentially towards zero with a time constant R_2C_2 as the condenser discharges through R_2 , and in this sense the circuit is able to remember previous events.

would be obtained by counting for an infinite time. In fact, the probable error on such a count will be $0.67/\sqrt{N}$ counts, a percentage error of $67/\sqrt{N}\%$.^{*} Thus by counting for four times as long the probable percentage error is halved.

The statistical fluctuations of the reading of a counting-rate meter are more complex. Fundamentally the rate meter functions as in Fig. 4, a charge of q /photon being fed into the condenser C_2 , tending to raise the voltage across the condenser; this charge leaks away exponentially through the resistance R_2 , and the mean current is indicated by the milliammeter. The product RC (R in megohms and C in microfarads) is a measure of the "memory" of the circuit in seconds, and Schiff and Evans¹⁵ have shown that statistically the counting-rate meter functions as if it counted for a time $2RC$. Thus, for a counting rate of M counts/sec. the meter effectively registers $2MRC$ counts and will fluctuate about the mean reading with a probable error of $67/\sqrt{2MRC}\%$. For a given rate the statistical fluctuations can be made as small as necessary by increasing the value of RC (most commercial units provide a wide range of possible RC values). The use of a large time constant is thus desirable to obtain accurate and steady readings, but in practice an upper limit exists because the rate meter takes $5RC$ sec. to reach a reading within 1% of the true one, and for this reason a scaling circuit can give a more reliable indication of any change in a shorter time. In most applications time constants much greater than 5 sec. are impracticable. Some of the errors introduced by the use of a large time constant will be discussed later.

Applications of Geiger Counters

(a) Debye-Scherrer-Hull Spectrum

This method, which necessitates a polycrystalline specimen, is used extensively in metallurgical research. One common

^{*} It is important to note that this is not the maximum error. About 1 reading in 20 will have an error 3 times this, 1 in 200 an error 4 times, and 1 in 1200 an error 5 times, and thus a convenient measure of the maximum error is 4 times the probable error. The comparison of calculated and observed errors is a good method of checking the

form of geometry used consists of a cylindrical wire specimen, surrounded by a concentric film on which the diffraction pattern shows as a series of "lines". Most spectrometers in essence retain this geometry, a Geiger counter replacing the film. Since a narrow slit is placed in front of the counter, it can detect only radiation diffracted over a very narrow angular region and, if the entire diffraction pattern is to be obtained, provision must be made to rotate the counter into all angular positions. Most spectrometers[†] use focusing geometry in order to increase the intensity of the pattern, and thereby reduce the statistical scatter, and the usual form of this is shown in Fig. 5. The source of diverging X-rays may be either the focus of the X-ray set, or, for more accurate work, the focus of a bent-crystal monochromator as shown.

For general use the spectrometer is usually equipped with facilities for rotating the counter about the spectrometer axis at a constant angular speed, and the counter is used in conjunction with a rate meter and an automatic recorder; the resulting chart gives a plot of the intensity against Bragg angle. An accuracy similar to that possible with films is obtained, but in most cases the required results can be obtained much more quickly. In cases where high accuracy is essential a scaling unit is used, the counter being set at specific angular positions and the intensity recorded for each position. Such a procedure is tedious and slow, but can yield results of a much higher accuracy than is possible by any other method. In all applications, using either a rate meter or direct counting, where high accuracy is required, the X-ray-tube output must be kept constant, or the variations must be measured

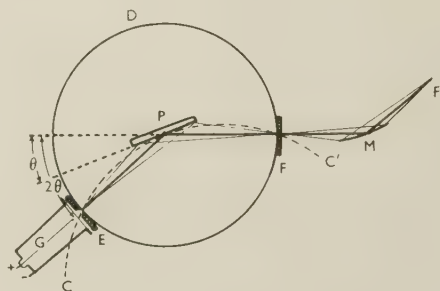


FIG. 5.—The Geometry of a Focusing Spectrometer. A narrow source of X-rays at F , consisting of either the line focus of an X-ray tube or the focus of a curved crystal monochromator M , as shown, diverges on to a specimen P at the centre of the spectrometer. The Geiger counter G can be rotated to any angle 2θ about this centre and detects rays passing through the slit E on the circle DEF . The chord EF subtends an angle $180-2\theta^\circ$ on all points on the circle CPC' and would thus give perfect focusing for a specimen conforming to this circle. The use of a flat tangential specimen such as P , gives negligible aberration if the divergence of the beam is less than 5° .

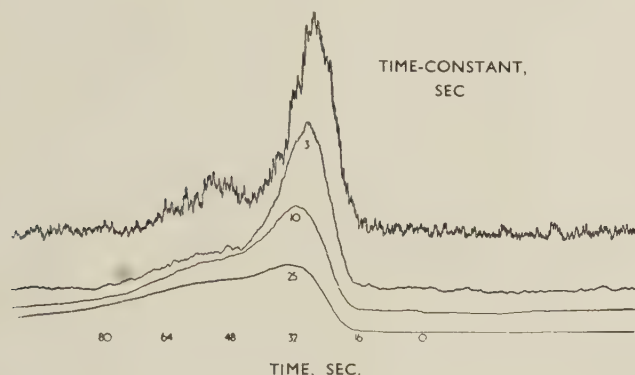
and a corrector applied. Hand control of the X-ray tube is very effective, but frequently inconvenient; Harris²² and Lang¹⁴ have standardized all readings against the tube current, and details of many electronic stabilizers have been published, but the most satisfactory method is direct monitoring of the X-ray output in which the ratio of the reflected intensity to the incident intensity is recorded (Hall, Arndt, and Smith¹⁸; Hall and Williamson²³).

reliability of the apparatus.

[†] The first spectrometer (Le Galley¹⁶) used a cylindrical specimen. Later models (Friedman¹⁷; Hall, Arndt, and Smith¹⁸) have used focusing of the type discussed by Bragg,¹⁹ Brentano,²⁰ and Brindley and Spiers.²¹

(b) Qualitative and Semi-Quantitative Chemical Analysis

Spectrometers using a counting-rate meter and automatic recording are excellent for applications in chemical analysis, since they may be made to give a direct record of intensity versus θ . Up to 1000 counts/sec. (Fig. 3) the response is approximately linear, and thus the approximate composition



[Courtesy Institute of Physics]

FIG. 6.—The Effect of Time Constants on X-Ray Lines (Wainwright²⁴).

An increase in the time constant reduces the statistical scatter, but results in a displacement of the apparent peak positions and, if used with too fast scanning rates, may smooth out the weaker lines.

of the sample may be determined from the peak intensity. The results may be used with an X-ray-diffraction data index in the usual way, starting with the stronger lines and eliminating possible components with the weaker lines. The results are such an improvement on those obtained with films, particularly from the point of view of intensities,* that future data cards will probably all be compiled from Geiger-counter studies.

The counter method, since it gives an almost instantaneous indication of intensity at a particular setting, offers many short cuts for high-speed analysis. This is frequently desirable, since a complete scan of the spectrum over the range $\theta = 10^\circ$ – 80° takes about the same time as the exposure of a fast film. If the possible constituents are known, or if some of the possibilities have been determined by a short, fast scan from 10° to 20° , analysis is very much quicker, as a system of searching by hand-scanning over a range of 1° about the expected Bragg angles can be used to determine which of the possible constituents is present. Such a "search" technique has been employed by Wainwright²⁴ to determine whether a series of samples were nickel- or chromium-plated. Wainwright also recommends that the initial scan for the strong lines should be carried out by hand in the interests of speed. Semi-quantitative analysis can be carried out simply, if the constituent phases are known but the relative amounts are not known, by calibrating the spectrometer with a series of known samples and graphing the values of the peak intensities against composition. An accuracy of $\pm 5\%$ in the measurement of intensities is good when a rate

meter is used; some early claims to much greater accuracy have been based on the peak counting rate, and have failed to take into account the loss of accuracy due to background level,[†] dead-time losses (Carl,²⁵ Lonsdale³³), circuit and incident X-ray intensity changes (Hall and Williamson²³ found a drift of $\pm 2\%$ even when using monitoring).

Detection of low-intensity lines due to impurities is difficult using a counting-rate meter, since a small time constant is necessary if the scan is to be fast, but this leads to a large statistical scatter which may mask small lines due to impurities. Wainwright²⁴ has investigated certain of the effects of time constants, and some of his results are shown in Fig. 6. With a small time constant (top curve) two peaks are evident, but the range of the statistical fluctuations is of the same order as the maximum value of the small peak. Increasing the time constant results in smaller fluctuations, but unless the speed of scanning is reduced it may involve loss of the weaker lines owing to the excessive smoothing action, as shown in the bottom curve of Fig. 6. Choice of correct time constant is very important, and in general it is preferable to err on the lower values. Wainwright recommends that fluctuations from the mean greater than 4 times the probable error should be re-investigated. Such re-investigation should preferably be carried out manually with a scaling unit, and this can be done immediately without disturbing the rest of the equipment. The potentialities of the method are very high, as is indicated in Fig. 7, which shows the diffraction pattern of tungsten powder annealed with a small quantity of steel filings.²⁷ The quantity of iron

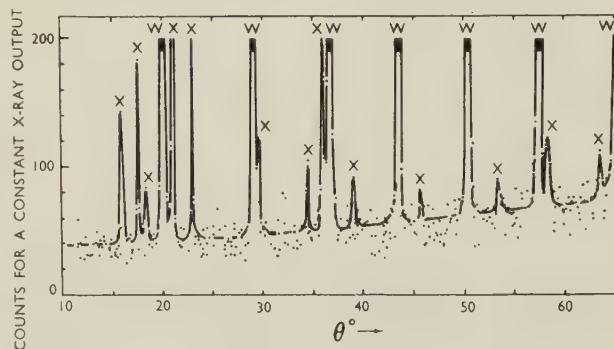


FIG. 7.—A Plot of the Background Level in the Spectrum of Tungsten Containing Less than 2% Impurity. The weak lines X are due to the impurity; those marked W are due to the tungsten and have peak intensities of about 10,000 counts. These results were obtained using monochromatic radiation, a scaling unit and continuous monitoring.

present is less than 2%, but a large number of lines are readily discernible which are due to the complex compounds formed. The extreme sensitivity of the method may be judged from the fact that the peak intensities of the main lines are about 15,000 counts, but it is unlikely that such sensitivity would be possible without the use of a monochromator.

* This improvement may be a source of embarrassment, since lines which are certainly the strongest have sometimes been classed as second- or third-strongest on the cards when films have been used. This danger is particularly great if the specimens for the data index were enclosed in thin tubes.

† The background level has a large effect since the squares of the errors add (Strong²⁶). Thus the apparent probable percentage error

in R readings of the peak intensity M superimposed on a background intensity m is $67/\sqrt{R(M+m)}$. The probable percentage error in the peak intensity reading however is $67\sqrt{1 + [(1 + R/P)(m/M)]}/\sqrt{RM}$, where P readings of the background intensity are taken. Thus if $M = m$, $R = 10$, and $P = 1$, the true error is 5 times greater than the apparent error.

(c) *Quantitative Analysis*

Analysis corresponding to an accuracy in the measurement of integrated intensities of better than $\pm 2\%$ is possible although extremely tedious (Hall and Williamson²³). Accuracies in the determination of composition corresponding to $\pm 5\%$ in intensity for strong lines and $\pm 10\%$ for weaker lines, using a counting-rate meter, can be achieved only if stringent precautions are taken at every stage (see, e.g., Lonsdale³³). Some of the early claims were over-optimistic but most workers now appear to obtain accuracies equivalent to those suggested above. The technique is particularly suited to ceramics and minerals (e.g. Tuttle and Cook³⁴; McCreery³⁵) and it has been applied to the determination of metal carbides by Redmond.³⁶ Heal and Mykura³⁷ have used a high-temperature camera to follow the transformation of austenite to martensite.

(d) *Parameter Studies*

The record of intensity against a calibrated scale of θ would make the Geiger-counter spectrometer seem ideal for accurate parameter studies, and, because of the high speed with which line position can be measured by a "search" technique, it

it is in this field that Geiger-counter spectrometers have already proved their superiority over film methods. Geometrics which avoid the necessity for an absorption correction over most of or all the pole figure, have been devised by Norton,²⁹ Schulz,³⁰ and Chernock and Beck.³¹ The ease with which the intensity, representing the density of the poles, may be read at a given setting allows the construction of a pole figure as the results are obtained. Two or three contour lines on a fairly fine grid of not more than 5° spacing may be constructed in an hour or two. Harris²² obtained an inverse pole figure directly from one scan of the spectrum of uranium. Recent studies of textures by Hu, Beck, and Sperry³² have shown the great accuracy possible in Geiger-counter determination of pole figures. It is quite feasible that such methods could be made sufficiently fast to be of value in the routine testing of the intermediate products in rolling mills, where the control of preferred orientation is of commercial value.

(f) *Other Applications*

Geiger counters have not yet been employed for many other X-ray studies. Cauchois used a Geiger-counter

TABLE I.—*Relative Merits of Methods of Detecting X-Rays.*

Ideal Features	Method	Films	Ion Chamber	Geiger Counter		Proportional Counter
				Counting-Rate Meter	Scaling Unit	
(1) Directness of intensity reading		Bad	Good	Good	Good	Good
(2) Large range of linear response		Fair	Good	Fair	Good *	V. good *
(3) High sensitivity		Bad	Bad	Fair	V. good	†
(4) Simple auxiliary equipment and handling		Fair-good	Fair	Fair	Bad	V. bad ‡
(5) Possible accuracy of intensity measurements		$\pm 5\%$ Bad-fair	$\pm 5\%$ Fair-good	$\pm 5\%$ Fair	$\pm 1\%$	$\pm 1\%$
(6) Stability: (a) with simplest equipment		Good	Good	Fair	Fair	Bad
(b) with best equipment		Good	Good	Good	V. good	Good
(7) Speed		Fair	Bad	Good	Bad	†

* A range of 0-1000 counts/sec. for Geiger counters, if counting-loss corrections are applied; 0 to 10^5 counts/sec. is possible with the proportional counters without correction.

† As for Geiger counter.

‡ The complexity of the auxiliary equipment necessary before a proportional counter can give even qualitative results is its principal disadvantage compared with a Geiger counter.

appears to be a very valuable tool for rapid phase-diagram determination. Unfortunately, two sources of error exist, one due to the counting-rate meter, and the other to the specimen. Wainwright²⁴ has demonstrated very convincingly (Fig. 6) how the use of a counting-rate meter shifts the lines owing to its memory effect. The peak of the line always coincides with a point of inflection, but this is not very satisfactory for accurate determinations. Scanning in both directions (increasing and decreasing θ) and taking the mean value, appears to be a better solution than very slow scanning speeds. The second difficulty is due to the fact that measurements of line position are not readily possible on both sides of the beam, and thus the surface of the specimen must be positioned very accurately, and the spectrometer zero carefully standardized. These errors have been discussed by Wilson,²⁸ but very careful work will be needed if the results are to be reliable, just as in the case of films.

(e) *Preferred Orientations*

The accuracy with which a counting-rate meter indicates intensity is adequate for studies of preferred orientation, and

spectrograph for analysing the fluorescent X-radiation from slags, and was able to detect $10^{-3}\%$ zinc with an accuracy of $10^{-4}\%$. Many workers have used them for lining-up fine collimators. Lonsdale³⁸ and Wooster, Ramachandran, and Lang³⁹ have made use of them for studying the diffuse reflections from single crystals, although in many cases Geiger counters are unsuitable because of the extremely high intensities frequently obtained in single-crystal cameras. Proportional counters in which dead-time losses are negligible and with a linear response up to intensities of 10^7 counts/sec. are more likely to be successful in this field.

Summary

Table I attempts to summarize the good and bad points of the common methods of detecting and measuring X-ray intensities. It should be emphasized that none of the methods, except that employing films, is in general suitable for two-dimensional studies, as in the Laue method, unless some preparatory exploration has been done.

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POWDER METALLURGY IN METALLURGICAL RESEARCH

By W. R. PITKIN * and D. J. JONES,* B.Sc., A.I.M.

Introduction

It is not proposed to discuss the merits or demerits of powder metallurgy as compared with the more orthodox method of melting and casting, but to present a number of examples in which, from necessity or desirability, use has been made of this technique in studying the properties of certain metals and alloys.

Research metallurgists in the lamp and valve industries have accepted, in some cases without choice, the "powder" approach, and have employed this method over the past thirty years as a means of studying, in particular, the structural characteristics of the high-melting-point metals tungsten and molybdenum. The experience gained in this field, with particular reference to the development of the tungsten lamp filament, provides a valuable basis and approach to a consideration of the use of this technique in other fields. It is proposed, therefore, to discuss the subject under the three following headings: (1) tungsten for lamp filaments; (2) alloys for valve filaments; and (3) alloys having specific properties.

Tungsten for Lamp Filaments

To produce a lamp of high efficiency it is necessary that the filament should be substantially free from movement throughout its useful life. For example, Fig. 1 (a) and (b) show two tungsten filaments which have been burnt in lamps for over 1000 hr. at *c.* 2400° C., that is, at a temperature equal to about 70% of the melting temperature. In Fig. 1 (a), the filament has remained substantially unchanged, the light output

dropping only slightly over the period of test. The filament in Fig. 1 (b), however, shows substantial movement and the light output dropped more markedly. This instability may appear in a general way, as shown in Fig. 1 (b), or in a more

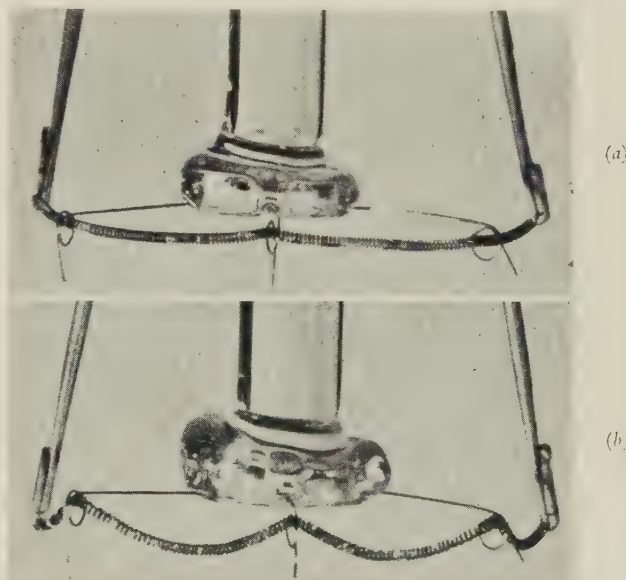


FIG. 1.—Stable (a) and Unstable (b) Tungsten Lamp Filaments.

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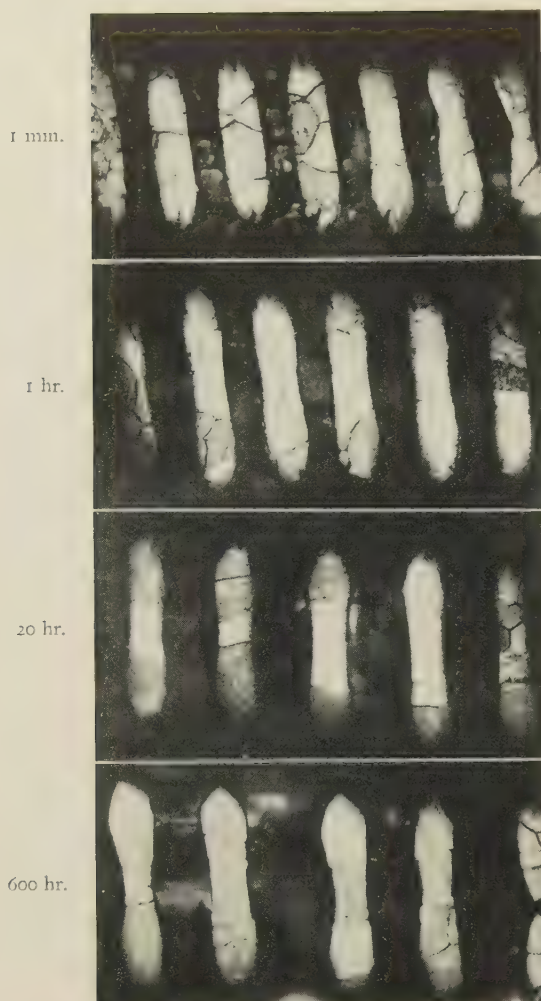


FIG. 2.—Effect of Heat-Treatment at $\approx 2400^{\circ}\text{C}$. on the Crystal Structure of a Very Pure Tungsten Filament.

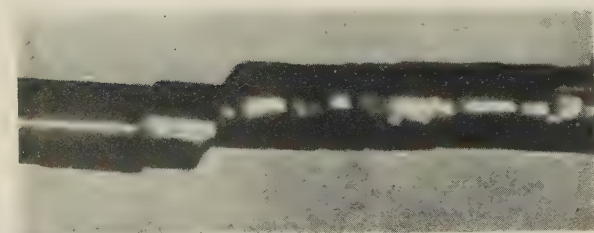


FIG. 3.—Offsetting Due to Grain-Boundary Movement in a Tungsten Filament.

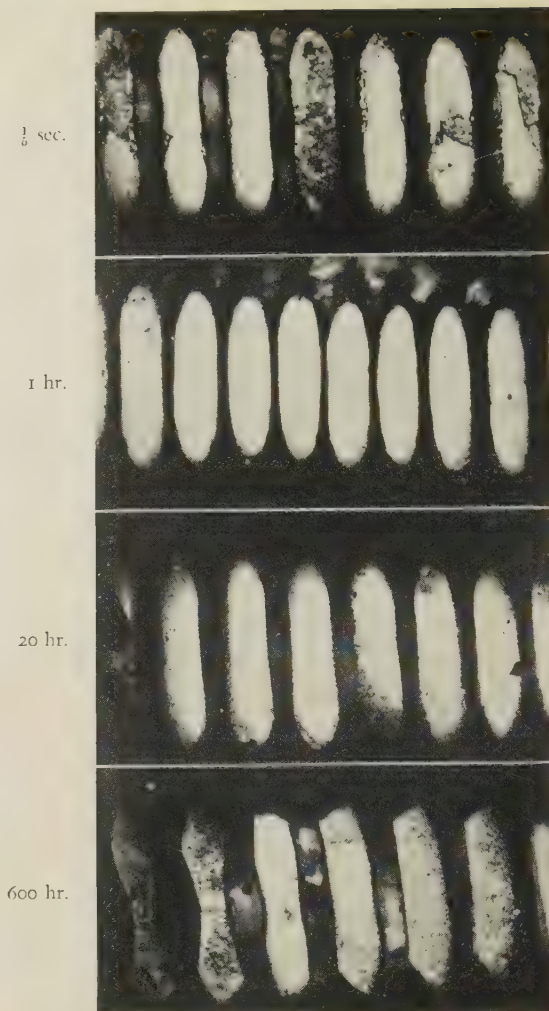


FIG. 4.—Stable Large-Grained Structure in Tungsten Containing Trace Additions of Alkali and Silica, After Heating at $\approx 2400^{\circ}\text{C}$. for Various Times.

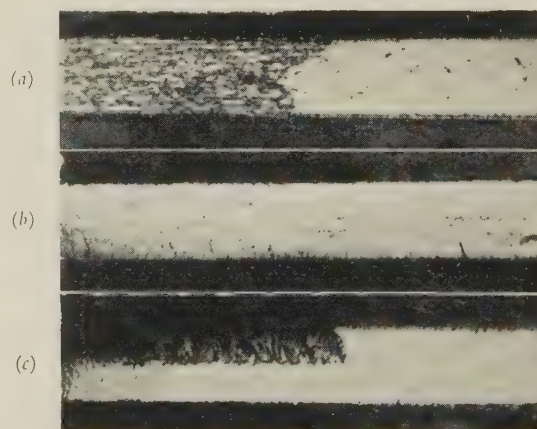


FIG. 5.—Types of Grain Boundary Obtained in Tungsten Wire by Controlled Additions. (a) Type at right angles to wire axis; (b) complete single crystals; (c) spliced or overlapping type.

specific manner such as localized distortion of turns or grain-boundary movement.

As a result of a study of the structure of filaments throughout their burning life, it became evident that instability was, in the main, associated with recrystallization and grain growth. For example, Fig. 2 illustrates the effect of heat-treatment at *c.* 2400° C. on the structure of a very pure tungsten filament. It is of interest to note the crystal size after 1 min. and the slight but progressive growth of these crystals throughout the time of 600 hr. A lamp filament with this type of structure would be unstable owing to the progressive grain growth throughout its life. Furthermore, apart from this

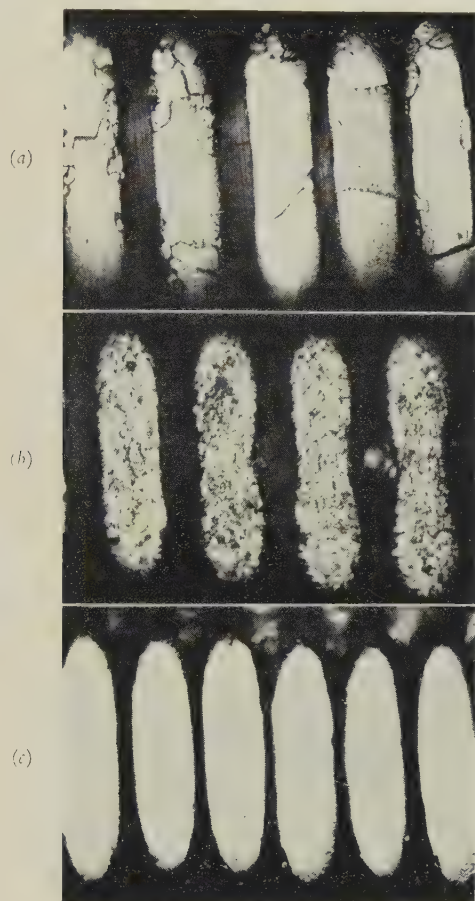


FIG. 6.—(a) Unstable Medium Grain-Size; (b) Stable Fine Grain-Size; (c) Stable Large Grain-Size in Tungsten Filaments, After Burning for 1000 hr. at *c.* 2400° C.

effect, trouble may arise owing to grain-boundary movement giving rise to offsetting, as shown in Fig. 3.

Thus, the problem resolved itself into the control of recrystallization and grain growth. The method of approach was two-fold: (i) production of a fine-grained structure in which further growth was inhibited; and (ii) production of a large-grained structure which remained stable from initial lighting of a filament to the end of its life.

These types of controlled structure are obtained by additions of refractory oxides. To produce a stable fine-grained structure it is usual to add approximately 0.5% thorium; on the other hand exaggerated growth is obtained by the addition of traces of, for example, alkali and silica.

Fig. 4 shows the stable exaggerated grain of tungsten containing traces of alkali and silica after heating for various times at *c.* 2400° C. Of particular interest is the almost instantaneous completion of grain growth on first heating to this temperature. This results in a very stable filament.

Reference has been made above to offsetting, which is associated with the existence of crystal boundaries running across a section of a wire, as seen in Fig. 2. To a certain extent, by suitably modifying the additions, some degree of control over the type of boundary may be maintained. For example, in Fig. 5 varying types of boundaries are shown. Of particular interest is the spliced or overlapping type; movement across or along this type of boundary is extremely difficult.

Thus, methods are available, using the powder technique, whereby the structure can be readily controlled. This technique is most convenient, as the addition of refractory oxides, either in the form of a suspension or as a solution of



FIG. 7.—Structure of (a) Ni-0.25% Al Alloy and (b) Ni-0.4% Al-0.2% Al_2O_3 Alloy, Annealed in Dry Hydrogen at 1000° C.

a suitable salt added to the oxide or to the metal powder, is relatively simple.

Fig. 6 shows the three typical structures of tungsten filaments referred to, after burning for 1000 hr. at *c.* 2400° C. These are, respectively, the unstable medium grain-size (a), the stable fine grain-size (b), and the stable large grain-size (c).

It is worth noting that, apart from the thorium addition, differences in purity of the basic tungsten cannot as yet be detected. This illustrates the important role played by minute impurities, suggesting that the powder technique can usefully be applied to a study of the effect of trace additions to other metals and alloys.

Alloys for Valve Filaments

This is a problem very similar to that already discussed, in which it is necessary to ensure very close control of composition and structural stability. Both these factors are highly important with respect to the thermionic and life characteristics of the valve. As the filaments in a valve are operated at elevated temperatures under a constant load, it is essential that they possess a relatively high resistance to creep.

In this field the powder technique has proved of real value

in studying the effect of metallic and non-metallic additions. For example, Fig. 7 shows the effect on the structure of an addition of alumina to a nickel-aluminium alloy. In this particular case, the aluminium in solution in the nickel raises the recrystallization temperature, and the presence of alumina restrains the grain growth, thus preventing offsetting.

In the preparation of alloys of this type, it is undesirable to mix aluminium powder with the nickel powder owing to the readiness with which aluminium oxidizes. The pro-

(a) *Investigation of a combination of metals of limited solubility and of widely differing melting points.*—The example chosen is a tungsten-nickel-copper alloy with a tungsten content of about 90%. Owing to the high melting point of tungsten, the powder method of approach proved invaluable. In this particular instance it was necessary to achieve a high density, coupled with specific mechanical properties. This entailed a study of part of the ternary system and of the effect of diffusion on the mechanical properties. Fig. 8 illustrates

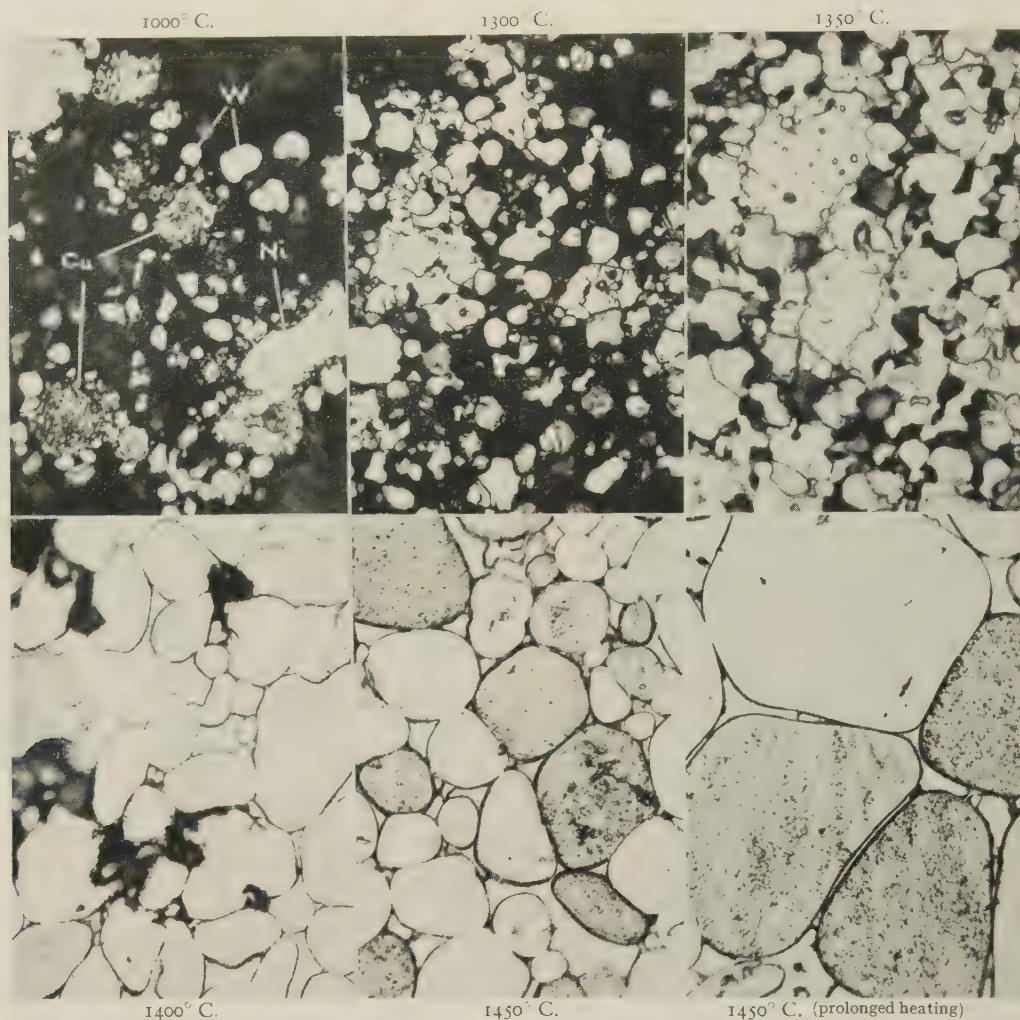


FIG. 8.—Effect of Temperature on the Progress of Sintering in a W-Ni-Cu Alloy. $\times 300$.

cedure adopted to overcome this difficulty consists of preparing a brittle nickel-aluminium alloy of approximately 50:50 composition by melting the metals in a purified hydrogen atmosphere and allowing the melt to freeze in the crucible. It is crushed and finally ball-milled to a fine powder. This fine powder is then mixed with the nickel powder in the required proportions. The method is particularly useful for all metals that oxidize readily.

Alloys Having Specific Properties

In this section three typical examples are discussed to illustrate the usefulness of the powder technique.

the effect of temperature on the progress of sintering in this system, the reactions consisting of the production of a nickel-copper liquid phase, solution of fine tungsten particles in this phase to saturation, then re-precipitation of tungsten on to existing tungsten grains.

(b) *Examination of the expansion characteristics of nickel-iron and similar alloys.*—In the electrical industry a range of nickel-iron alloys are used for sealing to glass, the tolerances on expansion being of the utmost importance. For some specialized applications it has been necessary to determine the expansion characteristics of very pure nickel-iron alloys. The powder technique provides a ready means of carrying out this type of investigation and in particular of obtaining

information on the effect of metallic impurities, which can be controlled to very close limits. The results have shown that it is possible by this method to manufacture such alloys with very close tolerances on expansion. This is of particular importance in specialized types of glass-to-metal seals.

(c) *The study of the properties of metals that are completely immiscible.*—For many purposes a combination of metals

completely immiscible in each other is of practical importance, e.g. copper-tungsten and silver-tungsten electrical contacts. The preparation of materials of this type is readily accomplished by the powder method, and the technique promises to be extremely useful for future work in this interesting field, particularly with regard to metallic and non-metallic combinations.

SOME METHODS OF MEASURING SURFACE TOPOGRAPHY AS APPLIED TO STRETCHER-STRAIN MARKINGS ON METAL SHEET

By W. H. L. HOOPER,* B.Sc., A.I.M., and J. HOLDEN,† Ph.D.

STRETCHER-strain markings can be very troublesome to users of sheet metal in pressing and other manipulative processes, but while light-reflection effects may be strong, giving the impression of deep surface distortions (Fig. 1) the differences

or aluminium alloy as soon as the yield point is reached, and others continue to appear during stretching up to an elongation of about 2%, beyond which strain markings are of relatively slight industrial significance.

Mechanical Measurements

Fig. 2 shows the set-up of the Talysurf exploring head in the examination of a stretched specimen which is securely bolted to a steel table. The head is lowered until the stylus,

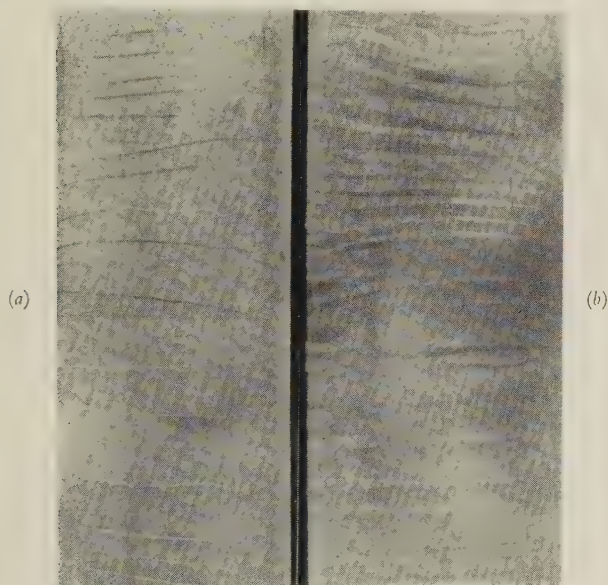


FIG. 1.—Stretcher-Strain Markings in (a) Steel and (b) Aluminium-Magnesium Alloy Pressings Made in the Same Die. $\times \frac{1}{4}$.

in level are, in fact, quite small. Two techniques for measuring the surface topography of sheet metal showing strain markings are described below, the first using the Talysurf surface-measuring instrument, and the second, light-interference effects.

The methods are illustrated by reference to parallel-sided strip specimens of dead-soft mild steel and aluminium-3% magnesium alloy, both of which develop strain markings of very similar superficial appearance when stretched on a tensile-testing machine. The first markings appear in either steel

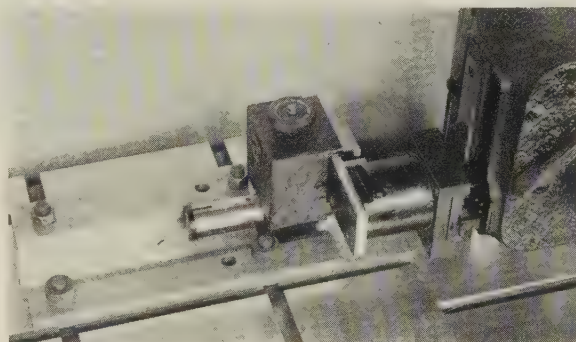


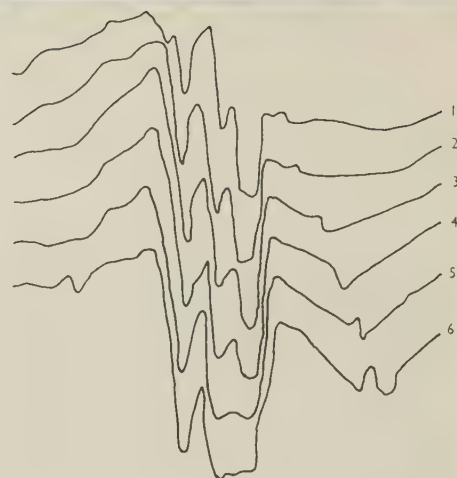
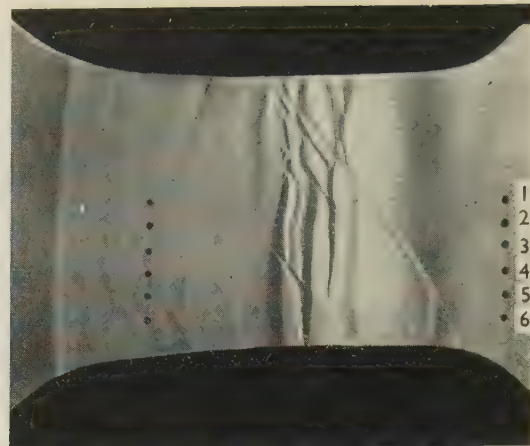
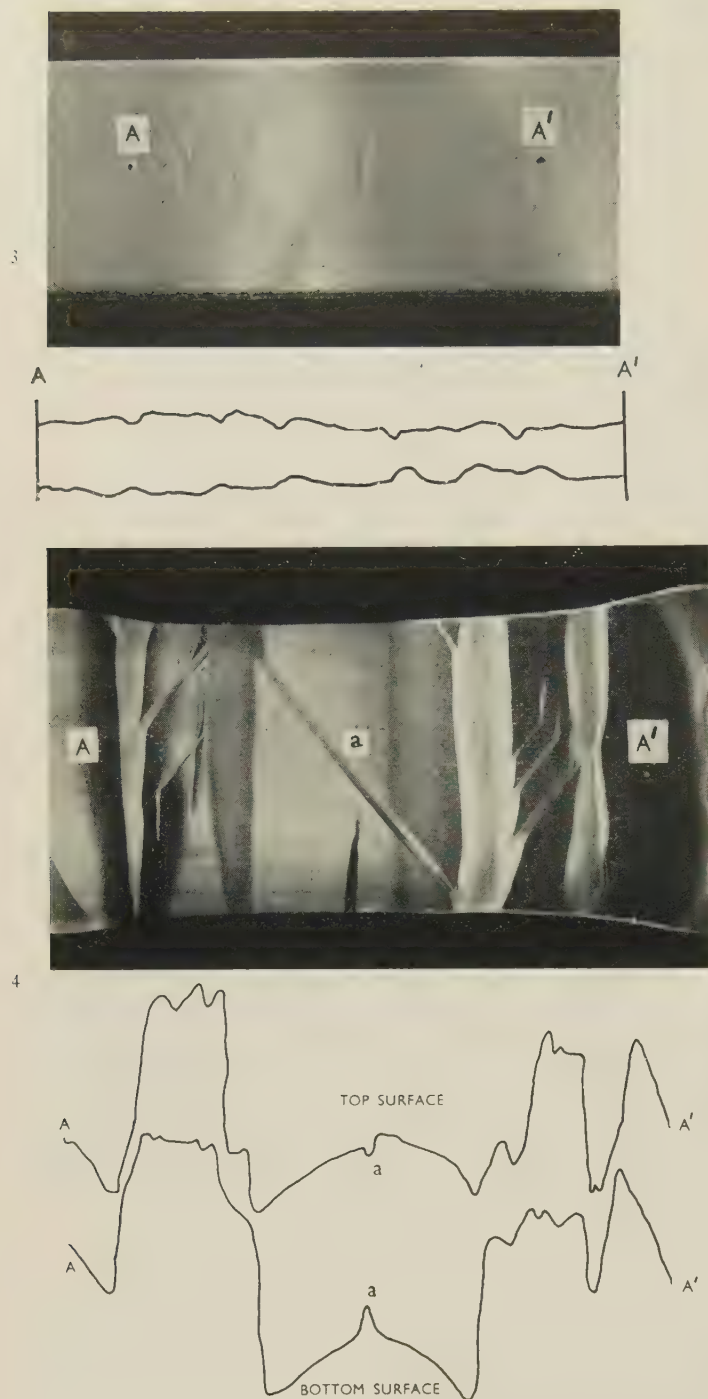
FIG. 2.—Exploring Head of Talysurf Instrument Arranged for the Measurement of Strain Markings.

tipped with a diamond of 0.0001 in. radius, makes contact with the surface under examination, and the table and specimen are then driven at a uniform speed in the right-to-left direction. The rise and fall of the stylus as it travels over the specimen surface are magnified electronically, the amplification chosen in this instance being $\times 1000$, and these magnified displacements are traced by a high-speed autographic recorder, the horizontal magnification in the present instance being $\times 2$.

Traces are taken from both surfaces of the specimen between two fixed points, and a typical example of the appearance of a steel specimen stretched $\frac{1}{2}\%$, and the record taken at this stage of deformation, are illustrated in Fig. 3. Jevons¹ has described stretcher-strain markings in steel as consisting, in their most typical form, of coincident depressions in opposite

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FIGS. 3-5.—Stretcher-Strain Markings in Steel Stretched $\frac{1}{2}\%$ ($\times 1$) and Corresponding Talysurf Traces ($\times 1000$ vertical magnification).

FIG. 3.—Wedge-Shaped Markings Associated with Necking of the Sheet.

FIG. 4.—Wedge-Shaped Markings and Kinks, Present Together.

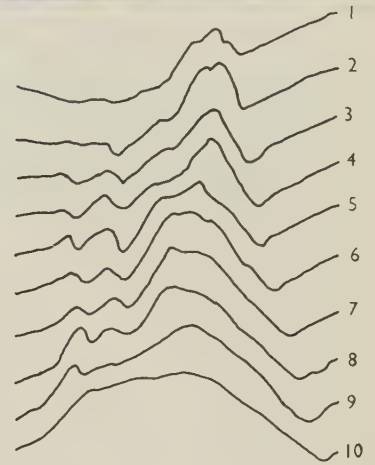
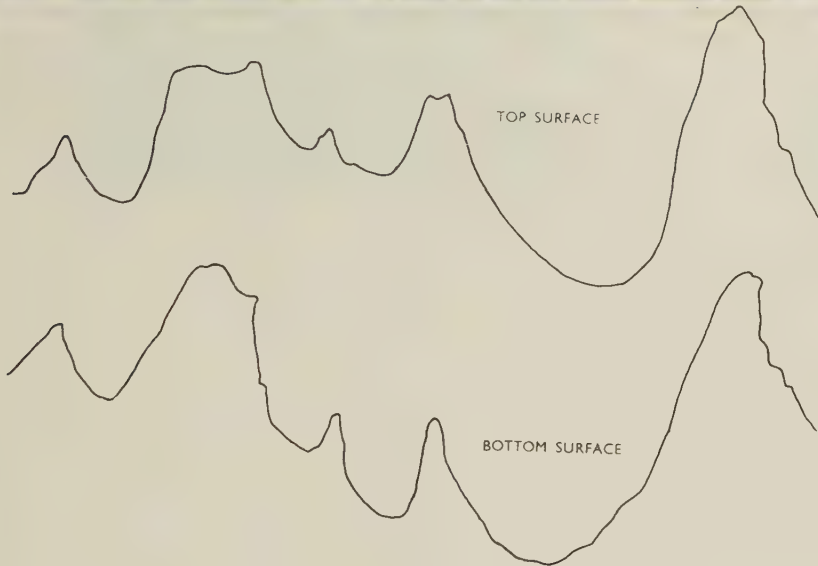
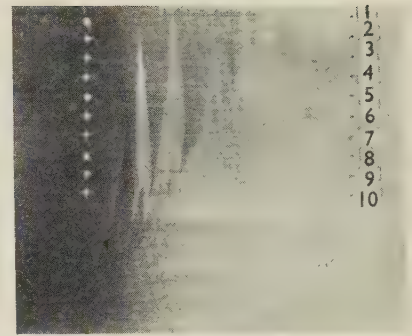
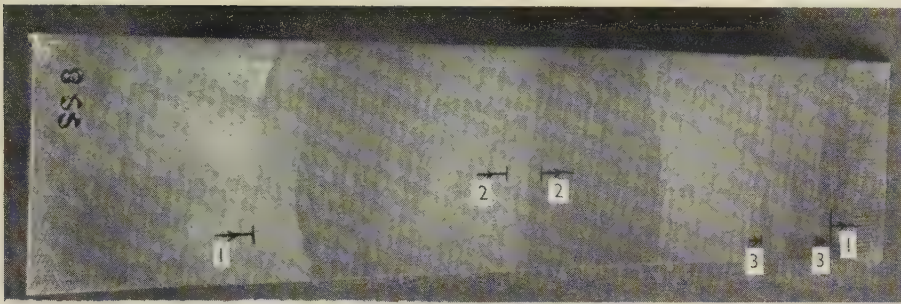
FIG. 5.—Talysurf Traces of a Small Area of One Surface.

sides of the sheet, so that a neck or wedge, associated with appreciable local thinning, is formed. The markings illustrated in Fig. 3 are of this type.

Stretched steel specimens may also display another type of marking, namely kinking, which is not associated with significant local thinning. In the specimen illustrated in Fig. 4, both kinks and necks are present, the kinks, in this instance, accounting for the most significant displacement and irregularity of the metal surfaces.

More detailed information on the physical form of stretcher-

strains is obtained by making a series of parallel traces over a small area of the stretched surface. The steel specimen shown in Fig. 5 was stretched $\frac{1}{2}\%$, and exhibits markings in various stages of development; the slight roughening of the left-hand half of the gauge-length has been caused by the spread of one group of markings, while a freshly formed group is evident in the middle of the gauge-length. Talysurf traces made at 0.1-in. intervals are shown as curves 1-6 and indicate that, when first formed, the markings consist of kinks and U- or V-shaped notches. The notch walls recede



FIGS. 6-7.—Stretcher-Strain Markings in Aluminium-3% Magnesium Alloy Stretched $\frac{1}{2}\%$ ($\times 1$) and Corresponding Talysurf Traces ($\times 1000$ vertical magnification).

FIG. 6.—Kink-Type Markings. The traces are between 1 and 1 on the photograph.

FIG. 7.—Talyurf Traces of a Small Area of One Surface.

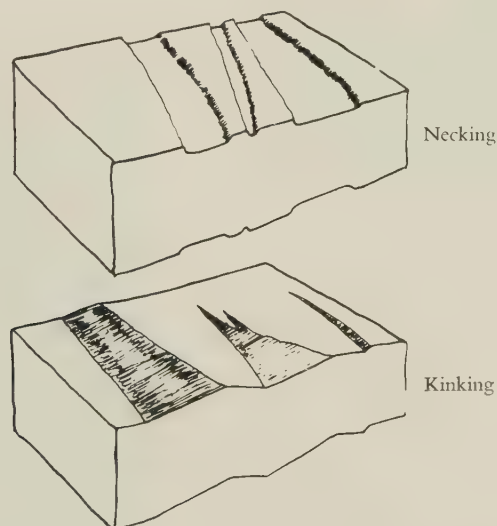
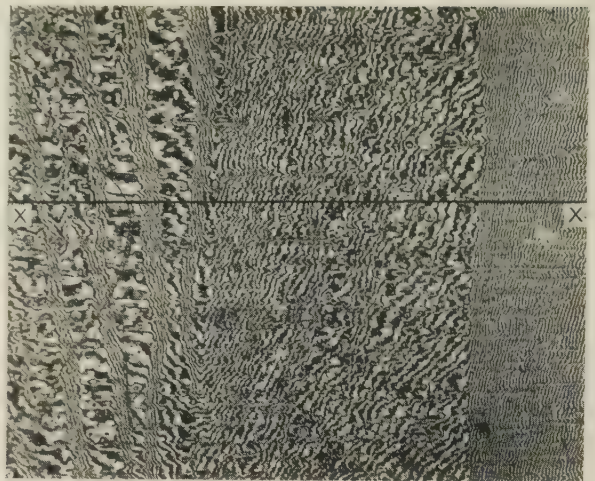
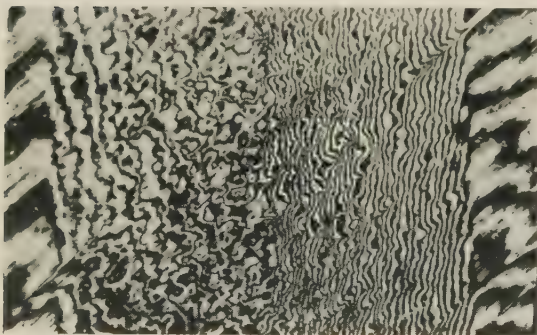
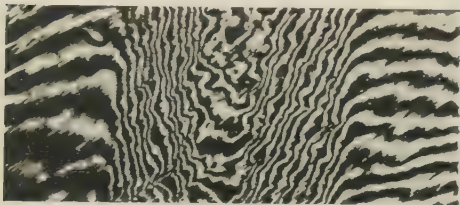
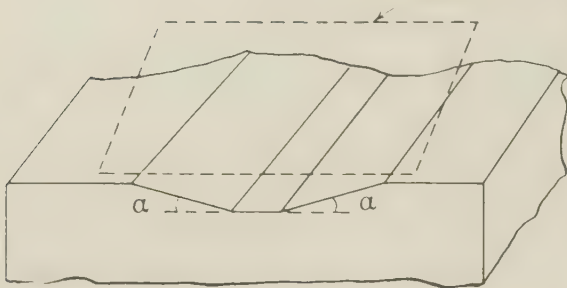


FIG. 8.—Diagrammatic Representation of Two Types of Marking Exaggerated with Respect to Sheet Thicknesses.

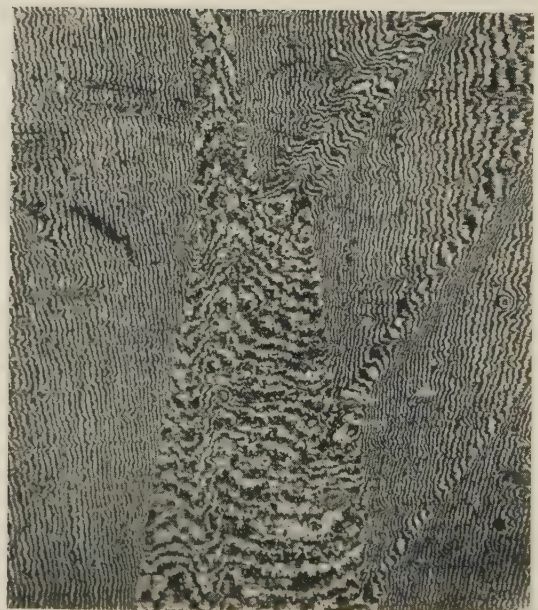
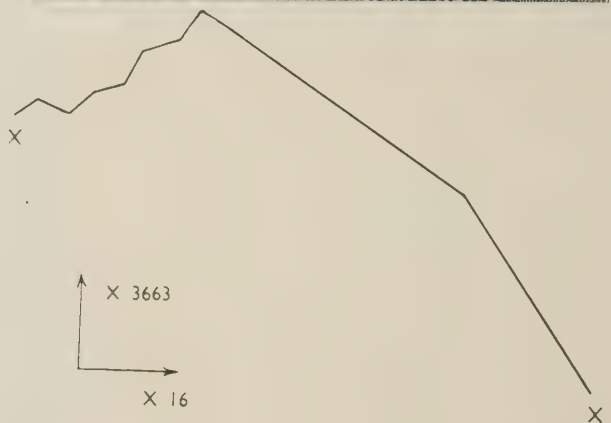
LOCAL SECTION SYMPOSIUM

INTERFEROMETRIC PATTERNS OF STRAIN MARKINGS.

GLASS REFERENCE SURFACE



11



12

FIGS. 9-10.—Channel-Marking in Steel (see "a", Fig. 4), with Sides at Equal Angles (Fig. 9, $\times 40$) and Different Angles (Fig. 10, $\times 90$) to the Undisturbed Surface.
FIG. 11.—Narrow Tongue-Like Markings (Kinks) in Aluminium-3% Magnesium Alloy. $\times 16$.
FIG. 12.—Complex Forked Marking in Aluminium-3% Magnesium Alloy. $\times 16$.

from each other as stretching progresses, so that flat-bottomed channels are formed. Between $\frac{1}{2}$ and 2% elongation the markings become more numerous but of diminished intensity. With more than about 2% extension they merge and the surface becomes comparatively smooth again.

Similar measurements have also been made of strain markings obtained by stretching annealed strips of aluminium-3% magnesium alloy sheet. The markings reach their greatest intensity when first formed, at the start of plastic deformation. The curves relating to the stretched aluminium specimen shown in Fig. 6 indicate that, at this stage, the markings consist of kinks, a peak on one surface being represented on the other by a valley, so that no thinning occurs. As stretching proceeds fresh groups form, and spread either by a smooth movement or by a more rapid shooting and branching, until at between 1 and 2% extension they merge, and thereafter rapidly diminish in intensity. No examples of local thinning have been observed in the examination of any aluminium specimens at small strains of the order of 0-2%, but it should be mentioned that at higher strains aluminium-magnesium alloys are subject to a further form of marking consisting of close parallel bands or necks, examples of which have been observed in Duralumin by Fell and others,²⁻⁴ while the contours of the markings in aluminium-3% magnesium alloy have been measured by the Talysurf technique by one of the present authors.⁵

More detailed examination of the small area shown in Fig. 7 indicates that adjacent areas of plane surface are inclined to each other at a slight angle, which calculation shows to be less than 1' relative to the undisturbed reference surface.

The characteristic form of the two types of observed markings, exaggerated in relation to the thickness of the material, are shown in perspective diagrams (Fig. 8). The maximum depth of markings measured in experiments on parallel-sided strips stretched on a tensile-testing machine was approximately the same in both steel and aluminium, i.e. about $\frac{3}{1000}$ in.

Interferometric Measurements

The principles of interferometry are well known and have been comprehensively described in the literature;^{6,7} consequently the discussion below is confined to a description of the technique employed in examining stretcher-strain markings.

The method consists, briefly, of covering the area of specimen under examination with a small piece of thin optical glass and illuminating the covered area by a parallel beam of monochromatic light, provided in this instance by a mercury-vapour lamp in conjunction with a filter which passes the mercury 5461 Å. green line. The specimen is viewed through a metallurgical microscope, modified by placing an extra iris diaphragm in the illumination arm; this does not involve any structural change to the microscope. For simple interferometric techniques the usual 16-mm. objective is satisfactory. Interference fringes are produced,

and their changes in separation and direction indicate the topography of the surface in the field of view. Interpretation of the fringe patterns is comparatively simple, and depths and angles of the surface markings can be accurately determined.

Fig. 9 is a photograph of a typical interference pattern caused by the diagonal marking in the steel specimen indicated as (a) on the Talysurf traces in Fig. 4. For ease of interpretation, the interference fringes have been arranged to run across the band at right angles, and the sketch in Fig. 9 indicates how this is effected by suitable placing of the glass reference surface. The fringe pattern then clearly indicates that the band consists of a groove with a narrow, flat bed and flat sides inclined at equal small angles to the undisturbed surface. The angle of inclination, obtained from counts of the average number of fringes in a given distance on the sloping channel side and on the undisturbed surface, ranges between 20' and 30'. Frequently the two sides of groove-like markings are inclined at different angles to the undisturbed surface, and this effect is illustrated by a typical photograph in Fig. 10.

An interference photograph of a group of narrow tongue-like markings in aluminium, similar to those shown in Fig. 7, is reproduced in Fig. 11. The exaggerated vertical section through the line XX in this figure, which was drawn from data calculated from the fringe spacing, is clearly similar to the Talysurf records shown in Fig. 7 and confirms the findings of the Talysurf method that, when first formed, strain markings in aluminium are flat, or slightly curved planes, orientated at a small angle to the undistorted surface.

Fig. 12 shows the more complex form of strain markings developed in aluminium at a later stage of stretching, by the shooting and branching of the original markings. It is clear however, that the characteristic flat inclined-plane formation of the markings is unchanged.

While confirming the measurements made upon stretcher-strain markings with the Talysurf instrument, the interference fringes, by illustrating the topography of an area instead of a line section, display the manner of termination and interconnection of individual bands; the complex branching deformations of "random markings" can also be studied in detail (see Fig. 12). In addition to the deformation bands themselves, it is seen that the fringes also illustrate the local surface undulations associated with the electropolishing of the surface, and the parallel scratches produced by the rolling of the specimens.

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AUTOGRAPHIC LOAD/STRAIN RECORDERS

By A. BINNS,* M.I.Mech.E.

Historical

THE use of autographic load/strain recorders with testing machines is quite a long-standing practice; they were applied to some of the earliest machines both of the single-lever and compound-lever types. Generally, the load was recorded by gearing a drum or flat chart-carrier to the poise-propelling gear, and the extension was transmitted from the grip holders by means of cords and sometimes by levers in addition. In some cases clips were fixed to the gauge-length of the specimen and the extension transmitted from this point, in order to eliminate the effect of slip in the grips and extension of the

accuracy. In one type of machine this was overcome by fitting a separate load-recording system, but this had the disadvantage that, being a separate system, careful calibration was necessary to ensure that the recorder agreed with the dial indicator.

The usefulness of the recorders mentioned was limited by the fact that the magnification of the extension of the specimen was not greater than about 20:1. With mechanical transmission it was not practicable to employ a higher ratio, on account of the degree of friction and inertia in the drum-and-pen mechanism, which necessitated a greater amount

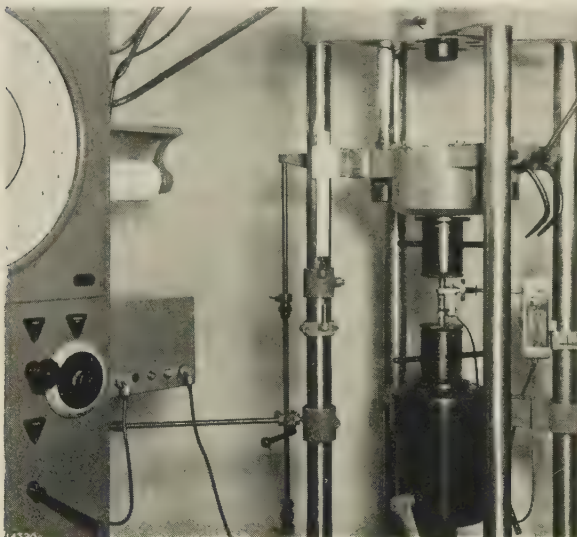


FIG. 1.—Automatic Stress/Strain Recorder Used in Conjunction with 30-Ton Testing Machine.

specimen beyond the gauge-length. The shape of the resultant curve depended to a large extent on the skill of the operator in so propelling the poise as to maintain the beam in equilibrium, and this form of recorder could not be considered as entirely satisfactory.

Wicksteed invented a recorder that was applied to Buckton machines, in which the beam was supported by a spring loaded by the setting of the poise at its maximum position. The effect of increased load on the test-piece was to reduce the pull on the spring and to cause linear movement of the beam with load; in other words, the machine was converted to an automatic spring balance. This device removed the necessity for skill on the part of the operator in propelling the poise, and consequently the records produced were very much better than on the earlier types of instrument.

Simple mechanical recorders were also fitted to dial machines, when these were introduced. One of the difficulties, again, was in connection with the load recording, as on some types of dial machine there was insufficient power in the dial mechanism to drive the pen recorder, so that the friction of this component exercised an adverse effect on the

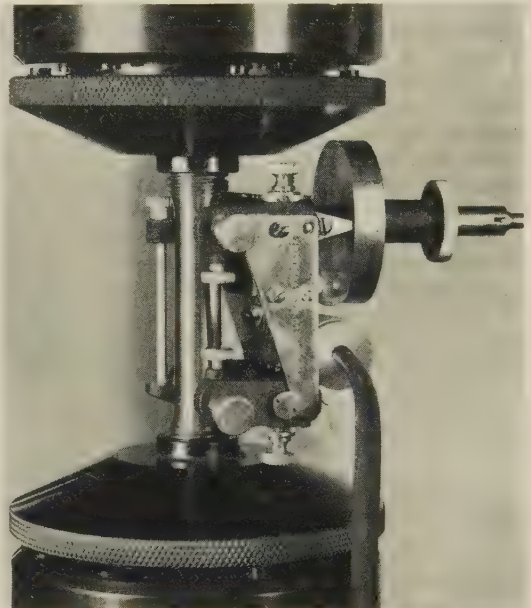


Fig. 2.—Hounsfield Extensometer Attached to Test-Piece.

of power than could be transmitted from the attachments to the gauge-length of the specimen. Above this ratio of magnification also the system became springy and subject to stretch of the cord, &c. However, mechanical recorders of this type and design, with magnifications up to 20:1, are manufactured to modern techniques and still find a useful application, particularly in technical colleges for teaching purposes and also by manufacturers interested in the behaviour of materials during the plastic stage, e.g. deep-drawn materials for press work.

The High-Magnification Recorder

The high-magnification recorder is a comparatively recent development. To be of use in exploring the elastic behaviour of a test-piece, the recorder should have a magnification of 200, 500, or even 1000:1, i.e. 1 in. on the diagram should be capable of representing 1/1000 in./in. of strain. With the development of modern electrical techniques, this problem has been solved.

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In the U.S.A. servo followers and electronic types are fairly well known, the most familiar perhaps being the Templin and Olsen "Atcotran". These mechanisms, however, are quite complicated, and although a large number have been made and seem to give satisfactory results in America, in this country we have not become quite so

of the recorder mechanism has no measurable effect on the accuracy of the indication.

Fig. 2 shows the extensometer, which is attached to the test-piece and is a standard Hounsfield type. This type of extensometer consists of two arms (A) and (B) fixed to the gauge-length of the test-piece (G) by means of spring-loaded

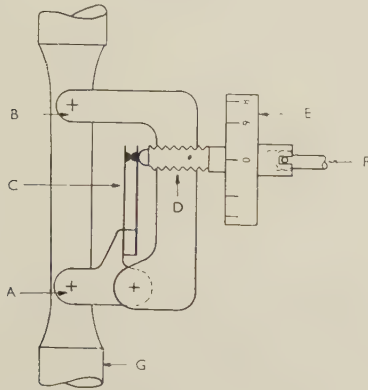


FIG. 3.—Hounsfield-Type Extensometer.

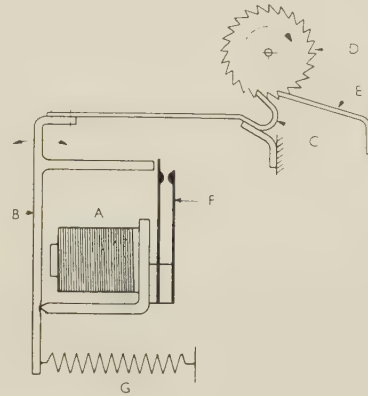


FIG. 4.—Extensometer Driving Unit.

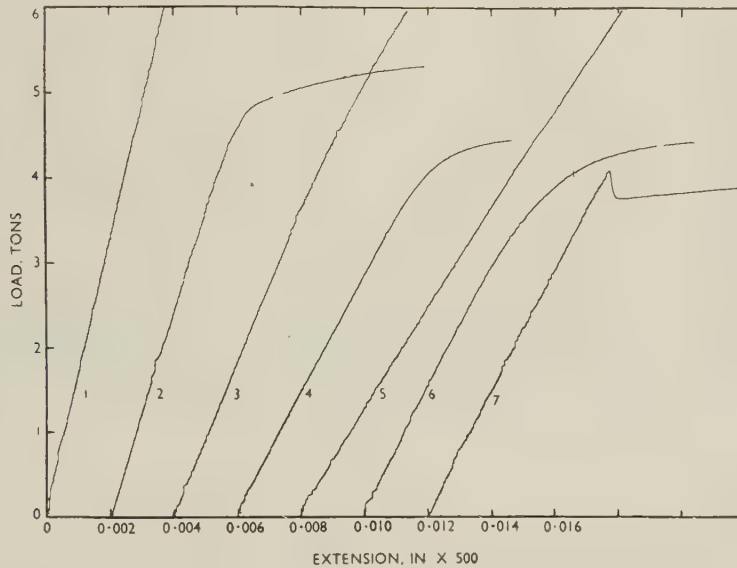


FIG. 5.—Specimen Graphs Obtained with Stress/Strain Recorder (2.00-In. Gauge-Length). Curves are displaced for clarity.

Curve 1.—0.564-in.-dia. Bright-Drawn Mild Steel. Curve 2.—0.500-in. dia. 12:12 Nickel-Chromium Stainless Steel. Curve 3.—0.564-in.-dia. Phosphor Bronze. Curve 4.—0.375-in.-dia. Bright-Drawn Mild Steel. Curve 5.—0.564-in.-dia. Duralumin. Curve 6.—0.500-in.-dia. Copper. Curve 7.—0.375-in.-dia. Annealed Mild Steel.

electronically minded, and the demand generally is for the simpler and more reliable methods.

The autographic stress/strain recorder now described is designed on this basis. It consists of five main units as shown in Fig. 1, which illustrates its application to a 30-ton-capacity machine. These are the drum-and-pen mechanism, the drum driving unit, the extensometer, the extensometer driving unit, and the power unit. The load ordinate is plotted automatically by the pen, which is coupled directly to the rack which rotates the pinion connected to the pointer of the dial indicator, and as this is driven by a large pendulum ample power is available for the purpose and the friction and inertia

cone pivots, and the one arm carries a pair of electric contacts (C) associated with a micrometer screw (D) on the other arm. This is shown diagrammatically in Fig. 3. The effect of extension of the test-piece is to open these contacts. Normally, a circuit is made through a battery and indicating lamp, so that immediately the test-piece extends, the light goes out. Contact is restored by rotating the micrometer drum (E). This is graduated, and accordingly a series of plots can be made as the test-piece extends. The sensitivity of the contacts is such that a movement of 1/50 of 1/1000 in. can be recorded. Instead of the micrometer drum being rotated by hand, the recorder does this automatically.

A diagrammatic representation of the extensometer driving unit is given in Fig. 4. It is very simple and consists essentially of an automatic telephone stepper switch. When energized, the magnet (A) draws the armature (B) towards it and moves the driving pawl (C) into the next tooth of the ratchet (D). The magnet is then de-energized and the spring return (G) drives the ratchet forward one tooth. The operation is similar to that which takes place when impulses are sent out from the dial of an automatic telephone. This piece of mechanism has been the subject of intensive development over many years and is mechanically safeguarded so that the ratchet cannot slip back and miss a tooth or travel only a portion of the tooth. The duty that it performs in the recorder is very light compared with the duty in an automatic telephone, and consequently the reliability and service life are very much extended.

A similar unit drives the drum of the recorder. It would not be possible to drive these stepper units directly from the micrometer contact on the extensometer, as both the current and the voltage across the contacts must be kept as low as practicable to avoid burning and welding. Accordingly, a power unit provides the necessary type of current and also the relays. The operation of the extensometer contacts controls a very sensitive Post-Office-type relay, which in turn controls a sequence relay equipped with a maintaining contact, so that once motion is initiated at least one complete step, or an equal whole number of steps, must be made on each unit before motion ceases. The extensometer driving unit always operates ahead of the drum driving unit and the normally open contact (F) on it then closes and supplies current to the drum driving unit. When the latter operates, its normally closed contact opens the maintaining circuit of the sequence relay and if, in the meantime, the micrometer screw

has advanced sufficiently to break the extensometer relay contact, motion ceases. If not, the complete cycle is repeated.

By this means the drum is rotated and the micrometer screw is advanced in synchronism by a series of equal pulses. These pulses produce a diagram in the form of a series of closely spaced steps either at 100 to the inch or 50 to the inch, in accordance with the magnification in use on the drum unit. The corresponding steps on the extensometer are 50 or 10 to 1/1000 in.

Fig. 5 shows some of the graphs produced by this instrument. It should be appreciated that each small step represents the extensometer plot, and while the appearance of the curves would be better if the steps were missing, the accuracy of the record would be no greater. The graphs shown are very early ones and contain one or two imperfections. Since they were made, experience has improved the technique, and perfect graphs can now be readily obtained. Such faults as there are in the graphs reproduced are not in the recorder, but are due to the extensometer and its attachment to the test-piece. It will be realized that in measuring to such fine limits, great care must be taken in regard to the attachment of the extensometer to the test-piece and to the axial alignment of the test-piece in the testing machine. The recorder will reproduce only what the extensometer tells it, and it is often found that the recorder diagnoses some hitherto unsuspected ailments, both in the test-pieces and the machines.

This recorder, by virtue of its simplicity and the ease with which high-magnification records can be made, should enable much more work to be done on the behaviour of materials during the elastic stage. It is believed also that it will facilitate considerably the obtaining of proof-stress results in routine test laboratories.

SPECIFICATION CREEP TESTING OF NIMONIC GAS-TURBINE ALLOYS

By C. W. WEAVER,* B.Sc., A.I.M.

Introduction

THE paper describes a machine developed to meet the growing demand for short-time creep tests, such as those required by D.T.D. Specifications 725, 736, and 747, on materials for gas-turbine rotor blading and other high-temperature applications. Compactness was important, combined with simplicity of design which would allow manufacture from commercial components, by makers not specialists in such equipment.

The machine, of the single-lever type and with a capacity of 1.5 tons, is arranged for continuous furnace operation, the test-piece and shackles being changed "hot". Compared with the usual procedure, this method reduces overshoot, simplifies control of temperature gradient, and, since the temperature is steady again in about 2 hr., saves considerable time.

The creep laboratory is situated in a basement, formerly an air-raid shelter, which after demolition of partition walls

gave a T-shaped area, with a "cross-bar" 52 × 18 ft. and an "upright" 36 × 24 ft., all with 7 ft. 6 in. head room. This accommodates 101 machines, with work benches, desks, storage cupboards, and maintenance gear. The design of the machine was governed by the limited space available and by the need to fit the units along the walls and between pillars. To save wall space, the electrical control panels are mounted on the machines, and, for simplicity, a mechanical thermostat is used for furnace-temperature control. A ventilation fan feeds 3000 ft.³ of air/min. through ducts near the ceiling.

A typical part of the installation is shown in Fig. 1.

Requirements of Routine Creep Testing

For routine testing a production-line approach was necessary, and the machine had to use the same test-piece and to work interchangeably with Denison T.46 radial machines already in service. It was designed to the following specification:

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(1) specimen size $1/20$ in.², 3-in. gauge-length, $1/2$ -in. Whitworth thread; (2) (a) load capacity $1\frac{1}{2}$ tons; (b) lever ratio 11:2:1; (3) temperature control $\pm\frac{1}{2}^{\circ}\text{C.}$; (4) furnace-temperature gradient 1°C. ; (5) height 6 ft.; (6) floor area 20×30 in.; (7) furnace size 12 in. dia. \times 15 in. long, hinged in a vertical line (this was the largest size of furnace possible); (8) shackle attachments by pin universal joints; (9) direct-straining screw attachment; (10) all-electrical control gear and thermostat apparatus mounted on the frame; (11) electric mains supplies fed by bus-bars from a tapped transformer, and the electrical supplies to the thermostats and time-counters similarly "piped" from a common 24-V. supply; (12) mechanical thermostat; (13) extensometer (i) for stress-rupture testing, a beam-operated dial gauge, (ii) for medium-sensitivity testing, a micrometer extensometer mounted on the specimen.

Two steel side plates, broached to take the three knife edges and joined by a welded steel stirrup, form the beam. Each beam is calibrated with its scale pan in a tensile machine, the pan and lever weight, made equivalent to 0.1-ton load, being adjusted to be exactly correct at the middle of the load range. The beams are accepted if the maximum error in $1\frac{1}{2}$ tons is within $\pm 0.3\%$.

Electrical Control Equipment

Each group of six machines has its own mains transformer for furnace supplies, and a 24-V. transformer and rectifier. The former has tapings at 110, 140, 170, 200, and 230 V. A.C., to bus-bars mounted in trunking and feeding small socket panels behind each machine, from which the voltage most appropriate to the operating temperature is selected. Another trunking contains two 24-V. D.C. lines, one for

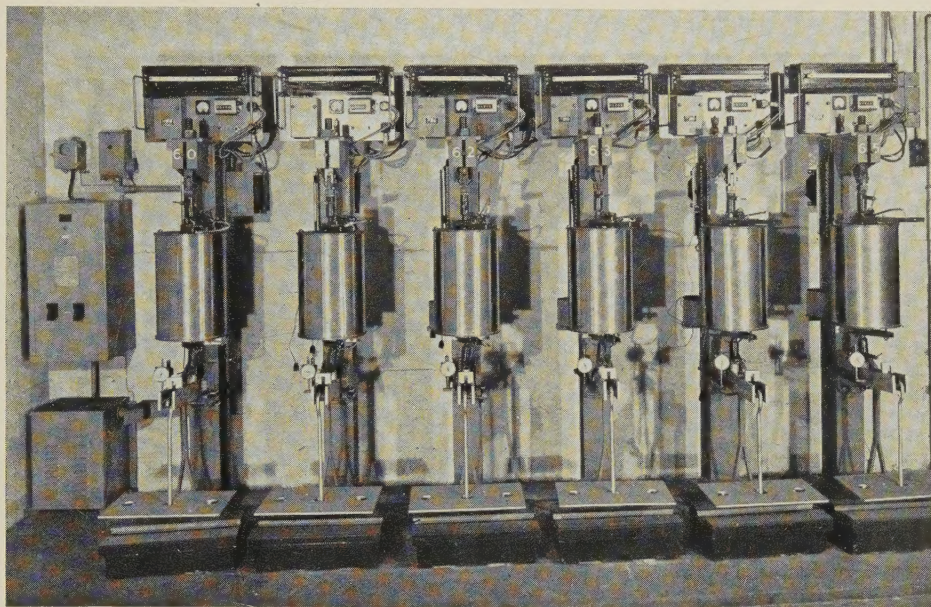


FIG. 1.—Group of Six Machines with Transformer, Rectifier, and Time Switch.

The specifications for Nimonic 80A and Nimonic 90 require tests of 75 hours' duration, at 17 and 19 tons/in.² respectively, in both cases at 750°C. Tests of long duration up to 900°C. are also required on experimental alloys.

Description of the Machine Frame

Fig. 2 shows the complete machine. The 8×6 in. I section *A* is welded to two 3×3 in. angle base members *B*, Rawl-bolted to the floor. At the top is welded a gusseted bearer-plate *C*, to support the shackle assembly, and a bracket *D* for the electrical panel. Lower down is bolted the loading-beam fulcrum *E*, and to the base angles is welded a support post. To relieve the test-piece of load, the beam is raised by a hand lever, with the top of this post as fulcrum, and is then supported by a peg inserted in the post. Furnace hinges and an extensometer bracket are also attached to the frame.

On the plate *C* a seating for the straining screw is drilled and pin-faced, and the fulcrum *E* is assembled and the machine levelled by a jig registering from the hole in this seating, to give axially of load on the test-piece.

the thermostat switch and relay circuits and the other for the hour-counters.

Connections are shown in Fig. 3 (*a*). The potentiometer rheostat *P* divides the current between the top and bottom furnace windings, which are in parallel. A choke *Q*, in series with the furnace, is short-circuited intermittently by a relay *R* operated by the mechanical thermostat switch *T*, so giving control by fluctuating input.

The hour-counter records to 999.9 hr. and is operated every 6 min. by a master time-switch common to six machines, the counters being wired in parallel. A manual switch *S*₂ and a micro-switch *S*₃, tripped by the beam when the specimen breaks, control each counter.

Connections to the control panel, except the neutral lead, are made by multi-pin plugs and sockets, the 24-V. supply being separate from the mains connections.

Construction of the Furnace

To provide an expansion member for the thermostat, a metal tube was necessary, and to give robustness and oxidation-resistance at high temperature, Inconel was chosen.

LOCAL SECTION SYMPOSIUM

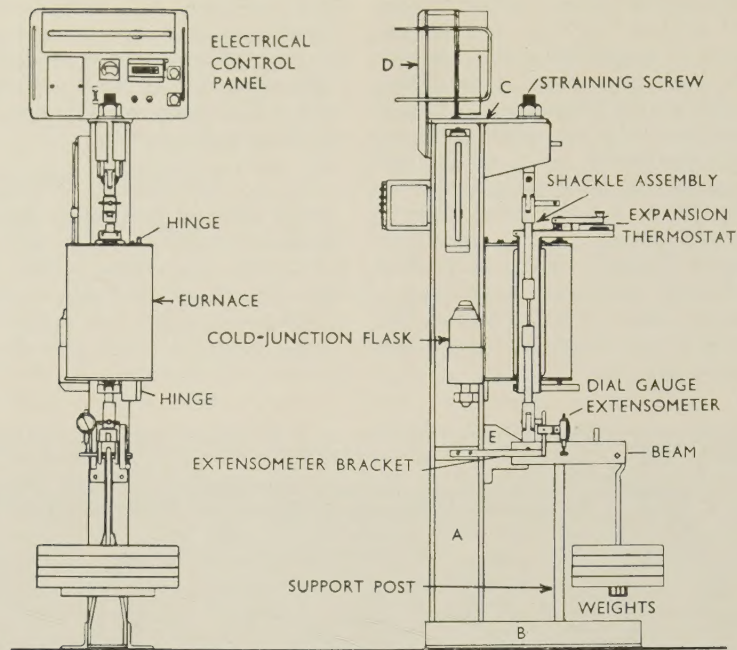


FIG. 2.—General Assembly of Stress-Rupture Creep Machines.

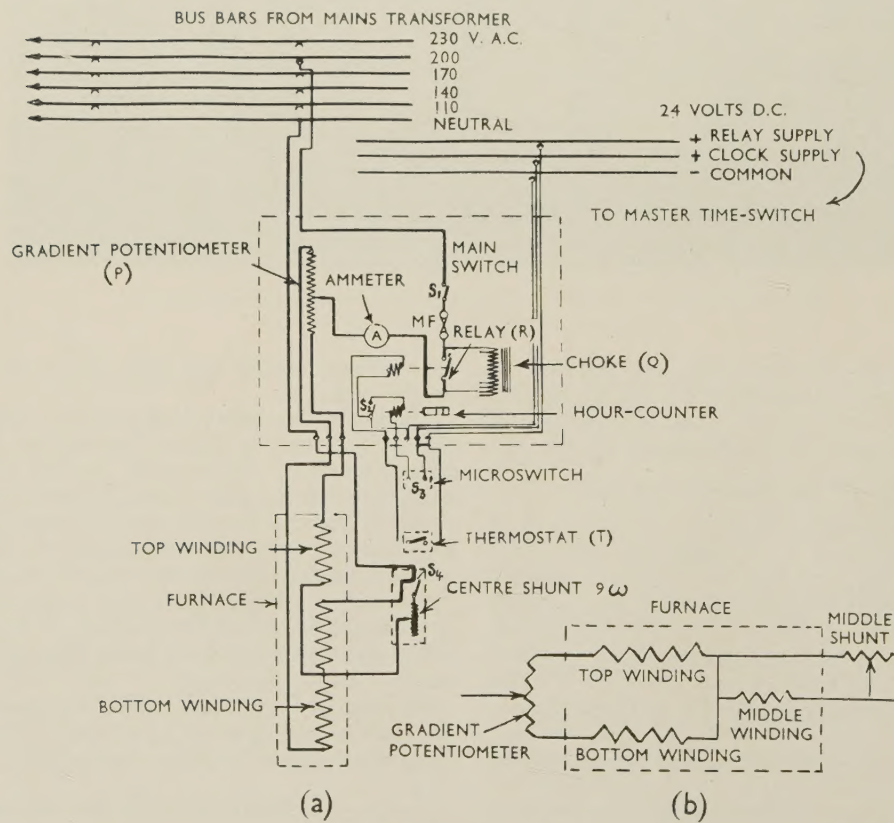


FIG. 3.—(a) Wiring Diagram. (b) Furnace-Circuit Diagram.

The circuit, Fig. 3 (b), with top and bottom heating windings in parallel, fed by the potentiometer rheostat *P*, permits wide variation of the relative currents without appreciable change of total input. The furnace-element construction is shown in Fig. 4. The tube is insulated, before winding, with $\frac{1}{8}$ in. of special alumina cement. The main windings are first spiralled on a $\frac{1}{4}$ -in. former to give the necessary high resistance in the limited length and each extends over $4\frac{1}{2}$ in., with a 4-in. gap between them. To trim the temperature at the middle of the test-piece, a small straight-wire winding is wound over the 3-in. middle section in series with the rest, and having a shunt resistance for adjustment. With the windings in place, insulating cement is brushed on, dried in successive layers and hardened by drying at 300°C ., baking for 4–5 hr. at 1000°C ., and then cooling to below 500°C ., when it forms a strong, hard white brick. The insulation should be 1–2 megohms at 500 V. The furnace leads are

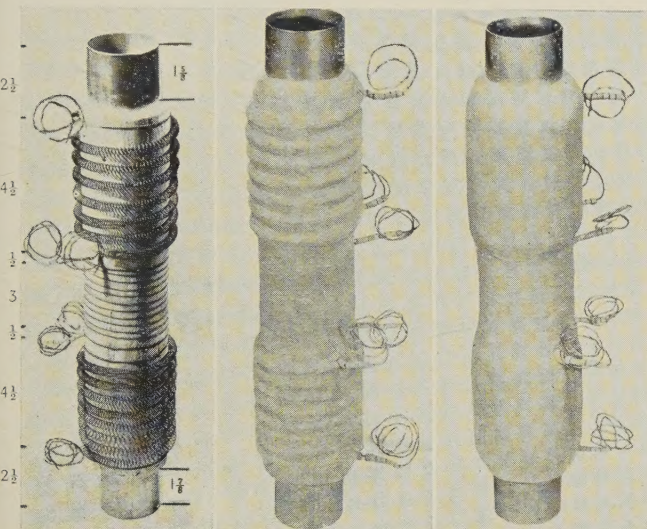


FIG. 4.—Details of Furnace-Tube Construction.

insulated with beads and are brought to terminals on the top plate near the hinge. The casing, which is made as large as practicable, is then packed with white diatomaceous earth, previously fired at 800°C . to drive off volatile carbonaceous material.

Temperature Control

The expansion controller is shown in Fig. 5. The Inconel furnace tube has two metal arms securely fixed to its ends which project beyond the casing. The lower arm carries a Nilo K* rod which passes up through the furnace body to the lever supported on the top arm, by which the difference in thermal expansion is magnified and transmitted to a small switch. The switch consists of two light levers in a moulded Bakelite box: one of these carries electrical contacts operating the 24-V. D.C. circuit which actuates the mains relay. The external lever is 10 : 1, and those in the box are 24 : 1, giving a magnification of 240 : 1. Coarse setting is by a 40 thread/in. screw bearing on the Nilo K rod, and fine adjustment is by a 40 thread/in. micrometer head at the end of the main lever. The temperature swing varies from $\pm\frac{1}{4}^{\circ}$ to $\pm\frac{1}{2}^{\circ}\text{C}$., and the mean temperature can be held to $\pm\frac{1}{2}^{\circ}\text{C}$. for long periods.

Operation of the Machine

The specimen, after measurement, is assembled in the shackles and adaptors, in a jig which fixes its position relative to the bottom shackle, thus controlling the vertical position in the furnace to facilitate temperature-gradient control.

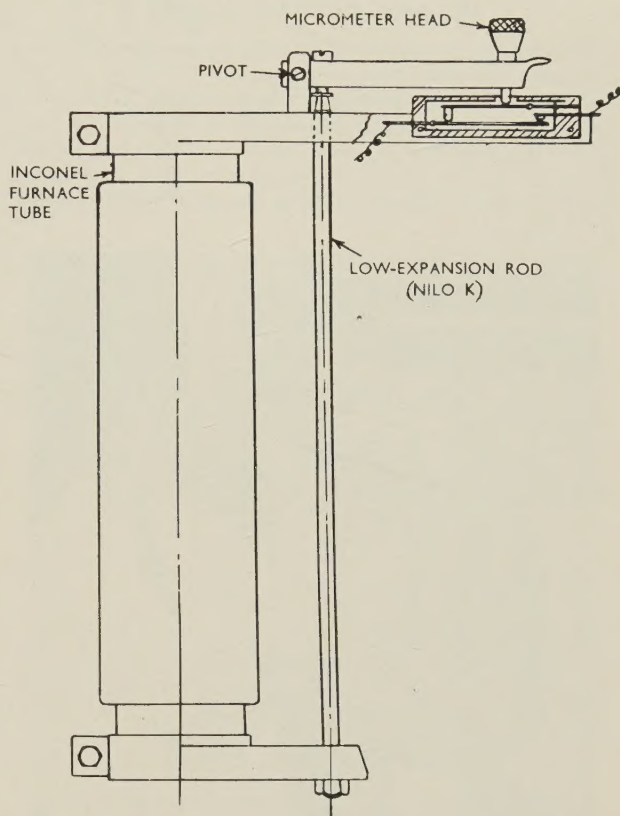


FIG. 5.—General Arrangement of Thermostat.

Thermocouples are tied on $\frac{1}{4}$ in. from the ends of the gauge-length, and the assembly is ready for insertion in the furnace. A third thermocouple can be tied at mid-length for gradient checking, but once the middle winding has been adjusted, this is found to remain steady. Fig. 6 shows the insertion into the furnace, the whole procedure being as follows: on conclusion of a test a pair of self-locking grips is fixed to the top shackle above the furnace. The beam and scale pan are lifted and pegged, using the long hand lever and the support post. The top and bottom shackle pins are then removed with a second pair of grips, so that the assembly is now supported by the top self-locking grip. The furnace is swung to the right, the packing is removed, and the assembly is lifted out from the top and placed to cool. If the bar is broken the bottom shackle must first be withdrawn downwards with the second pair of grips. The process is reversed to insert the new assembly. The thermocouples emerge at the top of the furnace. Platinum/platinum-10% rhodium thermocouples, 2 ft. 9 in. long, are used, with a thermos-flask cold junction on the side of the machine. At the cold junction, there are two-pin connectors to copper leads running to a multi-point potentiometer desk. Temperature is steady

* Nilo K is a Ni-Fe-Co controlled-expansion alloy, produced by Henry Wiggin and Co., Ltd.

within 2 hr., during which time gradient and overall adjustments are made. The test-piece can then be loaded, although in order to conform with the recommended "16-24 hr. at test temperature" of the British Standard Specification, the usual practice is to load next day.

Furnace-Gradient Control

Each furnace is calibrated with three thermocouples on a test-piece, the differences of temperature between top and middle, and bottom and middle thermocouples being plotted against the setting of the gradient potentiometer. The two curves will intersect at the point where there is no temperature difference between the top and bottom couples. The centre winding is then adjusted to bring the middle thermocouple

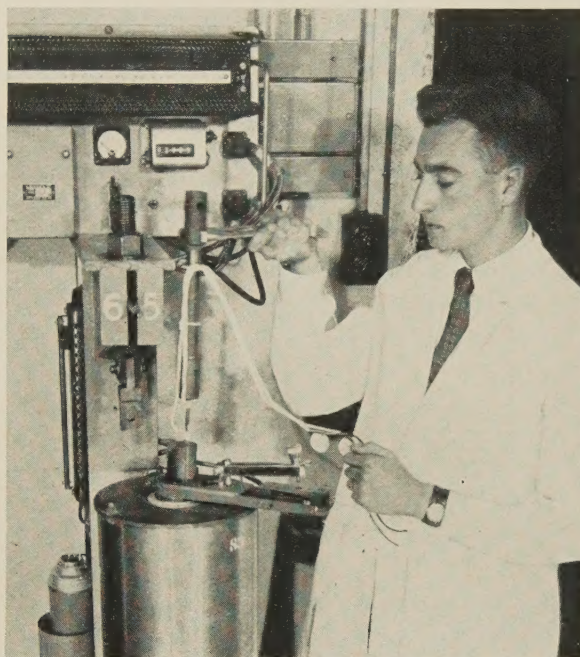


FIG. 6.—Method of Inserting Test-Piece into Hot Furnace.

to the same reading. After this adjustment fresh curves can be drawn, and it has been found that from test-piece to test-piece, provided the furnace position is constant, no subsequent adjustment of the centre winding is necessary. Then, in testing, if the top and bottom thermocouples show a gradient, the calibration curve shows how the gradient potentiometer needs to be adjusted.

A check with 12 couples along the 3-in. gauge-length, after careful adjustment, showed only $\pm 0.3^\circ \text{C}$. variation.

Extensometers

For the short-time specification and exploratory work, a dial gauge mounted on a bracket from the pillar A (Fig. 2)

records the movement of a horizontal lug on the beam 5 in. from the fulcrum. This gives a magnification of 5 times the extension, so that a 0.0005-in. division on the gauge equals 10^{-4} in. at the test-piece. Since such readings include extension and thermal expansion of shackles and adaptors, the apparent creep rate is slightly greater than the true rate. The nominal rate measurement is, however, adequate for production control and exploratory testing. To prevent damage to the gauge from thermal contraction if a furnace fails, the gauge is mounted on a pivot and held vertical by a spring clip, a short brass stop-sleeve being fitted to the plunger to limit the travel and then to transfer thrust from the mechanism to the body of the gauge. The thrust springs the gauge from the clip to swing on the pivot.

A micrometer extensometer for more precise reading has been adapted from that described by McKeown.¹ Two yokes screw on to the threads of the test-piece, and are locked by thin nuts. Rods from the top yoke pass down through tubes supported from the bottom yoke, and the relative movement is measured by 0.0001-in. ratchet micrometers fixed on the tubes. With this instrument a 6-in. test-piece with $\frac{3}{4}$ -in. Whitworth ends and a 3-in. gauge-length is used. Except for the micrometers, the instrument is made of special materials, yokes and lock nuts being in Nimonic 80, rods and tubes of Inconel, and micrometer mountings of low-expansion Nilo K.

Tests have been made with both the extensometer and the dial gauge in use on the same test-piece. In a test at 800°C . the results given by the two instruments are as follows:

Type of Extensometer	Creep Rate, $10^{-6}/\text{hr}$.		Total Creep, %		
	At 1000 Hr.	At 2000 Hr.	At 500 Hr.	At 2000 Hr.	At 3500 Hr.
Dial gauge	6.1	7.3	0.52	1.50	2.70
Micrometer	5.3	6.8	0.25	1.10	2.25

The main discrepancy occurs in the early stages, when the apparent primary stage is affected by the shackles and adaptors.

Two tests at 800°C ., on samples from the same bar, compared the micrometer extensometer in the machine described, with the Martens mirror-type in a high-sensitivity machine. Creep rates were low, the high-sensitivity test giving $2 \times 10^{-8}/\text{hr}$. at 2000 hr., compared with 4×10^{-8} for the stress-rupture machine, with total strains, at 2500 hr., of 0.026% and 0.037%, respectively.

Acknowledgements

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1. J. McKeown, *Metallurgia*, 1950, 42, 189.